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Response of RJ Lee Group to EPA Region 9 (Meer) Letter dated March 9, 2006

Regarding Evaluation of EPA's Analytical Data from the El Dorado Hills Asbestos Evaluation Project

Exhibit C *3.0*

Analytical Methods used for Soil Samples

Date: July 2006

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be controlled to prevent exposure of the public to potential health and safety hazards at the disposal site. Therefore, for liability protection of operators of landfills that handle asbestos, fencing and warning signs are recommended to control public access when natural barriers do not exist. Access to a landfill should be limited to one or two entrances with gates that can be locked when left unattended. Fencing should be installed around the perimeter of the disposal site in a manner adequate to deter access by the general public. Chain-link fencing, 6-ft high and topped with a barbed wire guard, should be used. More specific fencing requirements may be specified by local regulations. Warning signs should be displayed at all entrances and at intervals of 330 feet or less along the property line of the landfill or perimeter of the sections where asbestos waste is deposited. The sign should read as follows:

ASBESTOS WASTE DISPOSAL SITE
BREATHING ASBESTOS DUST MAY CAUSE LUNG DISEASE AND CANCER

Recordkeeping. For protection from liability, and considering possible future requirements for notification on disposal site deeds, a landfill owner should maintain documentation of the specific location and quantity of the buried asbestos wastes. In addition, the estimated depth of the waste below the surface should be recorded whenever a landfill section is closed. As mentioned previously, such information should be recorded in the land deed or other record along with a notice warning against excavation of the area.

[52 FR 41897, Oct. 30, 1987, as amended at 62 FR 1834, Jan. 14, 1997]

Appendix E to Subpart E of Part 763—Interim Method of the Determination of Asbestos in Bulk Insulation Samples

Section 1, Polarized Light Microscopy

1.1 Principle and Applicability

Bulk samples of building materials taken for asbestos identification are first examined for homogeneity and preliminary fiber identification at low magnification. Positive identification of suspect fibers is made by analysis of subsamples with the polarized light microscope.

The principles of optical mineralogy are well established.[1,2] A light microscope equipped with two polarizing filters is used to observe specific optical characteristics of a sample. The use of plane polarized light allows the determination of refractive indices along specific crystallographic axes. Morphology and color are also observed. A retardation plate is placed in the polarized light path for determination of the sign of elongation using orthoscopic illumination. Orientation of the two filters such that their vibration planes are perpendicular (crossed polars) allows observation of the birefringence and extinction characteristics of anisotropic particles.

Quantitative analysis involves the use of point counting. Point counting is a standard technique in petrography for determining the relative areas occupied by separate minerals in thin sections of rock. Background information on the use of point counting[2] and the interpretation of point count data[3] is available.

This method is applicable to all bulk samples of friable insulation materials submitted for identification and quantitation of asbestos components.

1.2 Range

The point counting method may be used for analysis of samples containing from 0 to 100 percent asbestos. The upper detection limit is 100 percent. The lower detection limit is less than 1 percent.

1.3 Interferences

Fibrous organic and inorganic constituents of bulk samples may interfere with the identification and quantitation of the asbestos mineral content. Spray-on binder materials may coat fibers and affect color or obscure optical characteristics to the extent of masking fiber identity. Fine particles of other materials may also adhere to fibers to an extent sufficient to cause confusion in identification. Procedures that may be used for the removal of interferences are presented in Section 1.7.2.2.

1.4 Precision and Accuracy

Adequate data for measuring the accuracy and precision of the method for samples with various matrices are not currently available. Data obtained for samples containing a single asbestos type in a simple matrix are available in the EPA report *Bulk Sample Analysis for Asbestos Content: Evaluation of the Tentative Method.*[4]

1.5 Apparatus

1.5.1 *Sample Analysis*

A low-power binocular microscope, preferably stereoscopic, is used to examine the bulk insulation sample as received.

- *Microscope:* binocular, 10–45X (approximate).
- *Light Source:* incandescent or fluorescent.
- *Forceps, Dissecting Needles, and Probes*
- *Glassine Paper or Clean Glass Plate*

Compound microscope requirements: A polarized light microscope complete with polarizer, analyzer, port for wave retardation plate, 360° graduated rotating stage, substage condenser, lamp, and lamp iris.

- *Polarized Light Microscope:* described above.

- *Objective Lenses:* 10X, 20X, and 40X or near equivalent.
- *Dispersion Staining Objective Lens* (optional)
- *Ocular Lens:* 10X minimum.
- *Eyepiece Reticle:* cross hair or 25 point Chalkley Point Array.
- *Compensator Plate:* 550 millimicron retardation.

1.5.2 *Sample Preparation*

Sample preparation apparatus requirements will depend upon the type of insulation sample under consideration. Various physical and/or chemical means may be employed for an adequate sample assessment.

- *Ventilated Hood* or negative pressure glove box.
- *Microscope Slides*
- *Coverslips*
- *Mortar and Pestle:* agate or porcelain. (optional)
- *Wylie Mill* (optional)
- *Beakers and Assorted Glassware* (optional)
- *Certrifuge* (optional)
- *Filtration apparatus* (optional)
- *Low temperature asher* (optional)

1.6 Reagents

1.6.1 *Sample Preparation*

- *Distilled Water (optional)*
- *Dilute CH_3COOH:* ACS reagent grade (optional)
- *Dilute HCl:* ACS reagent grade (optional)
- *Sodium metaphosphate* $(NaPO_3)_6$ (optional)

1.6.2 *Analytical Reagents*

Refractive Index Liquids: 1.490–1.570, 1.590–1.720 in increments of 0.002 or 0.004.

- *Refractive Index Liquids for Dispersion Staining:* high-dispersion series, 1.550, 1.605, 1.630 (optional).
- *UICC Asbestos Reference Sample Set:* Available from: UICC MRC Pneumoconiosis Unit, Llandough Hospital, Penarth, Glamorgan CF6 1XW, UK, and commercial distributors.
- *Tremolite-asbestos* (source to be determined)
- *Actinolite-asbestos* (source to be determined)

1.7 Procedures

NOTE: Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are usually friable and may release fibers during handling or matrix reduction steps. All sample and slide preparations should be carried out in a ventilated hood or glove box with continuous airflow (negative pressure). Handling of samples without these precautions may result in exposure of the analyst and contamination of samples by airborne fibers.

1.7.1 *Sampling*

Samples for analysis of asbestos content shall be taken in the manner prescribed in Reference 5 and information on design of sampling and analysis programs may be found in Reference 6. If there are any questions about the representative nature of the sample, another sample should be requested before proceeding with the analysis.

1.7.2 *Analysis*

1.7.2.1 *Gross Examination*

Bulk samples of building materials taken for the identification and quantitation of asbestos are first examined for homogeneity at low magnification with the aid of a stereomicroscope. The core sample may be examined in its container or carefully removed from the container onto a glassine transfer paper or clean glass plate. If possible, note is made of the top and bottom orientation. When discrete strata are identified, each is treated as a separate material so that fibers are first identified and quantified in that layer only, and then the results for each layer are combined to yield an estimate of asbestos content for the whole sample.

1.7.2.2 *Sample Preparation*

Bulk materials submitted for asbestos analysis involve a wide variety of matrix materials. Representative subsamples may not be readily obtainable by simple means in heterogeneous materials, and various steps may be required to alleviate the difficulties encountered. In most cases, however, the best preparation is made by using forceps to sample at several places from the bulk material. Forcep samples are immersed in a refractive index liquid on a microscope slide, teased apart, covered with a cover glass, and observed with the polarized light microscope.

Alternatively, attempts may be made to homogenize the sample or eliminate interferences before further characterization. The selection of appropriate procedures is dependent upon the samples encountered and personal preference. The following are presented as possible sample preparation steps.

A mortar and pestle can sometimes be used in the size reduction of soft or loosely bound materials though this may cause matting of some samples. Such samples may be reduced in a Wylie mill. Apparatus should be clean and extreme care exercised to avoid cross-contamination of samples. Periodic checks of the particle sizes should be made during the grinding operation so as to preserve any fiber bundles present in an identifiable form. These procedures are not recommended for samples that contain amphibole minerals or vermiculite. Grinding of amphiboles may result in the separation of fiber bundles or the production of cleavage fragments with aspect ratios greater than 3:1. Grinding of vermiculite may also produce fragments with aspect ratios greater than 3:1.

Acid treatment may occasionally be required to eliminate interferences. Calcium carbonate, gypsum, and bassanite (plaster) are frequently present in sprayed or trowelled insulations. These materials may be removed by treatment with warm dilute acetic acid. Warm dilute hydrochloric acid may also be used to remove the above materials. If acid treatment is required, wash the sample at least twice with distilled water, being careful not to lose the particulates during decanting steps. Centrifugation or filtration of the suspension will prevent significant fiber loss. The pore size of the filter should be 0.45 micron or less. Caution: prolonged acid contact with the sample may alter the optical characteristics of chrysotile fibers and should be avoided.

Coatings and binding materials adhering to fiber surfaces may also be removed by treatment with sodium metaphosphate.[7] Add 10 mL of 10g/L sodium metaphosphate solution to a small (0.1 to 0.5 mL) sample of bulk material in a 15-mL glass centrifuge tube. For approximately 15 seconds each, stir the mixture on a vortex mixer, place in an ultrasonic bath and then shake by hand. Repeat the series. Collect the dispersed solids by centrifugation at 1000 rpm for 5 minutes. Wash the sample three times by suspending in 10 mL distilled water and recentrifuging. After washing, resuspend the pellet in 5 mL distilled water, place a drop of the suspension on a microscope slide, and dry the slide at 110 °C.

In samples with a large portion of cellulosic or other organic fibers, it may be useful to ash part of the sample and view the residue. Ashing should be performed in a low temperature asher. Ashing may also be performed in a muffle furnace at temperatures of 500 °C or lower. Temperatures of 550 °C or higher will cause dehydroxylation of the asbestos minerals, resulting in changes of the refractive index and other key parameters. If a muffle furnace is to be used, the furnace thermostat should be checked and calibrated to ensure that samples will not be heated at temperatures greater than 550 °C.

Ashing and acid treatment of samples should not be used as standard procedures. In order to monitor possible changes in fiber characteristics, the material should be viewed microscopically before and after any sample preparation procedure. Use of these procedures on samples to be used for quantitation requires a correction for percent weight loss.

1.7.2.3 *Fiber Identification*

Positive identification of asbestos requires the determination of the following optical properties.

- Morphology
- Color and pleochroism
- Refractive indices
- Birefringence
- Extinction characteristics
- Sign of elongation

Table 1-1 lists the above properties for commercial asbestos fibers. Figure 1-1 presents a flow diagram of the examination procedure. Natural variations in the conditions under which deposits of asbestiform minerals are formed will occasionally produce exceptions to the published values and differences from the UICC standards. The sign of elongation is determined by use of the compensator plate and crossed polars. Refractive indices may be determined by the Becke line test. Alternatively, dispersion staining may be used. Inexperienced operators may find that the dispersion staining technique is more easily learned, and should consult Reference 9 for guidance. Central stop dispersion staining colors are presented in Table 1-2. Available high-dispersion (HD) liquids should be used.

TABLE 1-1—OPTICAL PROPERTIES OF ASBESTOC FIBERS

Mineral	Morphology, color[a]	Refrac- tive indices[b]		Birefringence	Extinction	Sign of elonation
		α	γ			
Chrysotile (asbestiform serpentine).	Wavy fibers. Fiber bundles have splayed ends and "kinks". Aspect ratio typically >10:1. Colorless[3], nonpleochroic.	1.493–1.560	1.517–1.562[f] (normally 1.556).	.008	∥ to fiber length.	+ (length slow)
Amosite (asbestiform grunerite).	Straight, rigid fibers. Aspect ratio typically >10:1. Colorless to brown, nonpleochroic or weakly so. Opaque inclusions may be present.	1.635–1.696	1.655–1.729[f] (normally 1.696–1.710.	.020–.033	∥ to fiber length.	+ (length slow)
Crocidolite (asbestiform Riebeckite).	Straight, rigid fibers. Thick fibers and bundles common, blue to purple-blue in color. Pleochroic. Birefringence is generally masked by blue color.	1.654–1.701	1.668–1.717[3e] (normally close to 1.700).	.014–.016	∥ to fiber length.	– (length fast)

TABLE 1–1—OPTICAL PROPERTIES OF ASBESTOC FIBERS—Continued

Mineral	Morphology, color[a]	Refrac- tive indices[b]		Birefringence	Extinction	Sign of elonation
		α	γ			
Anthophyllite-asbestos.	Straight fibers and acicular cleavage fragments.[d] Some composite fibers. Aspect ratio <10:1. Colorless to light brown.	1.596–1.652	1.615–1.676[f].	.019–.024	∥ to fiber length.	+ (length slow)
Tremolite-actinolite-asbestos.	Normally present as acicular or prismatic cleavage fragments.[d] Single crystals predominate, aspect ratio <10:1. Colorless to pale green.	1.599–1.668	1.622–1.688[f].	.023–.020	Oblique extinction, 10–20° for fragments. Composite fibers show∥ extinction.	+ (length slow)

[a] From reference 5; colors cited are seen by observation with plane polarized light.
[b] From references 5 and 8.
[c] Fibers subjected to heating may be brownish.
[d] Fibers defined as having aspect ratio >3:1.
[e] ∥ to fiber length.
[f] ⊥To fiber length.

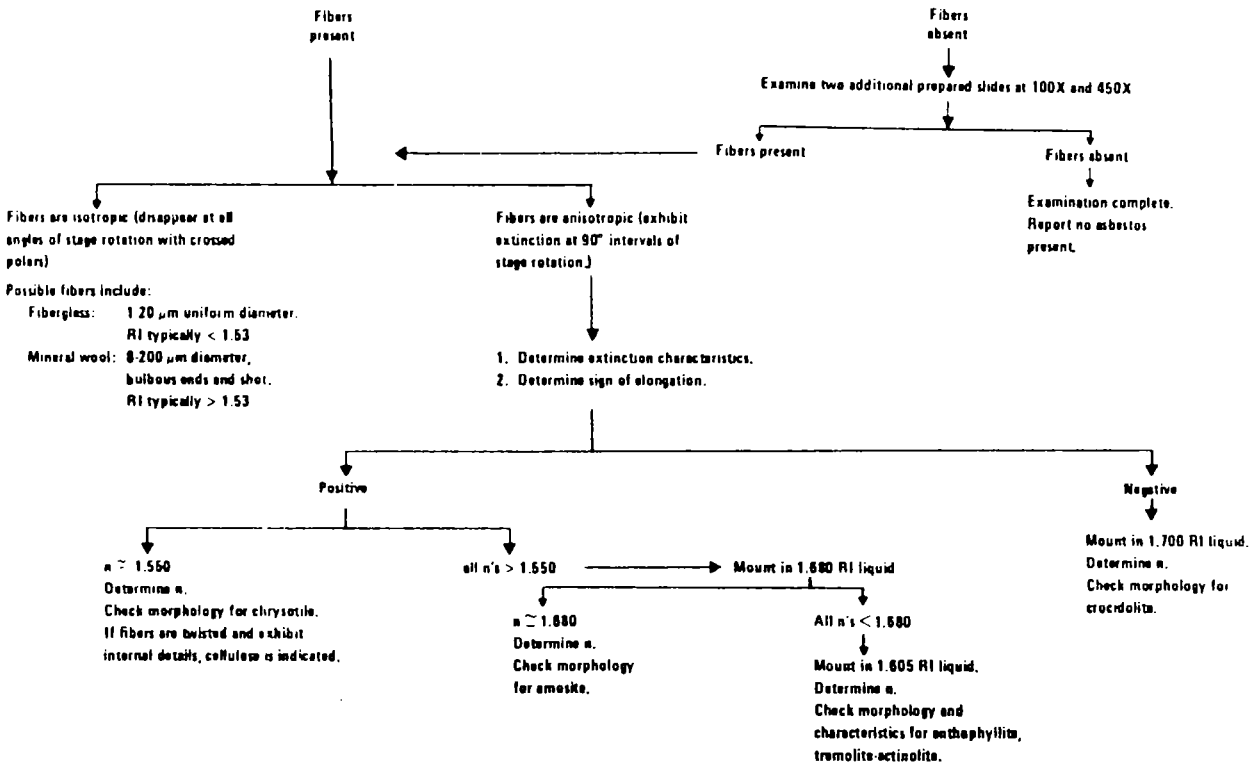

Figure 1-1. Flow chart for analysis of bulk samples by polarized light microscopy.

TABLE 1–2—CENTRAL STOP DISPERSION STAINING COLORS[A]

Mineral	RI Liquid	η	η⊥
Chrysotile	1.550[HD]	Blue	Blue-magenta
Amosite	1.680	Blue-magenta to pale blue.	Golden-yellow
	1.550[HD]	Yellow to white.	Yellow to white
Crocidolite[b]	1.700	Red magenta	Blue-magenta
	1.550[HD]	Yellow to white.	Yellow to white
Anthophyllite	1.605[HD]	Blue	Gold to gold-magenta
Tremolite	1.605[HD c]	Pale blue	Gold
Actinolite	1.605[HD]	Gold-magenta to blue.	Gold
	1.630[HD c]	Magenta	Golden-yellow

[a] From reference 9.
[b] Blue absorption color.
[c] Oblique extinction view.

1.7.2.4 *Quantitation of Asbestos Content*

Asbestos quantitation is performed by a point-counting procedure or an equivalent estimation method. An ocular reticle (cross-hair or point array) is used to visually superimpose a point or points on the microscope field of view. Record the number of points positioned directly above each kind of particle or fiber of interest. Score only points directly over asbestos fibers or nonasbestos matrix material. Do not score empty points for the closest particle. If an asbestos fiber and a matrix particle overlap so that a point is superimposed on their visual intersection, a point is scored for both categories. Point counting provides a determination of the area percent asbestos. Reliable conversion of area percent to percent of dry weight is not currently feasible unless the specific gravities and relative volumes of the materials are known.

For the purpose of this method, "asbestos fibers" are defined as having an aspect ratio greater than 3:1 and being positively identified as one of the minerals in Table 1-1.

A total of 400 points superimposed on either asbestos fibers or nonasbestos matrix material must be counted over at least eight different preparations of representative subsamples. Take eight forcep samples and mount each separately with the appropriate refractive index liquid. The preparation should not be heavily loaded. The sample should be uniformly dispersed to avoid overlapping particles and allow 25-50 percent empty area within the fields of view. Count 50 nonempty points on each preparation, using either

- A cross-hair reticle and mechanical stage; or
- A reticle with 25 points (Chalkley Point Array) and counting at least 2 randomly selected fields.

For samples with mixtures of isotropic and anisotropic materials present, viewing the sample with slightly uncrossed polars or the addition of the compensator plate to the polarized light path will allow simultaneous discrimination of both particle types. Quantitation should be performed at 100X or at the lowest magnification of the polarized light microscope that can effectively distinguish the sample components. Confirmation of the quantitation result by a second analyst on some percentage of analyzed samples should be used as standard quality control procedure.

The percent asbestos is calculated as follows:

$$\% \text{ asbestos} = (a/n)\ 100\%$$

where

a=number of asbestos counts,
n=number of nonempty points counted (400).

If a=0, report "No asbestos detected." If $0 < a \leq 3$, report "<1% asbestos".

The value reported should be rounded to the nearest percent.

1.8 References

1. Paul F. Kerr, *Optical Mineralogy,* 4th ed., New York, McGraw-Hill, 1977.

2. E. M. Chamot and C. W. Mason, *Handbook of Chemical Microscopy, Volume One,* 3rd ed., New York: John Wiley & Sons, 1958.

3. F. Chayes, *Petrographic Modal Analysis: An Elementary Statistical Appraisal,* New York: John Wiley & Sons, 1956.

4. E. P. Brantly, Jr., K. W. Gold, L. E. Myers, and D. E. Lentzen, *Bulk Sample Analysis for Asbestos Content: Evaluation of the Tentative Method,* U.S. Environmental Protection Agency, October 1981.

5. U.S. Environmental Protection Agency, *Asbestos-Containing Materials in School Buildings: A Guidance Document,* Parts 1 and 2, EPA/OPPT No. C00090, March 1979.

6. D. Lucas, T. Hartwell, and A. V. Rao, *Asbestos-Containing Materials in School Buildings: Guidance for Asbestos Analytical Programs,* EPA 560/13-80-017A, U.S. Environmental Protection Agency, December 1980, 96 pp.

7. D. H. Taylor and J. S. Bloom, Hexametaphosphate pretreatment of insulation samples for identification of fibrous constituents. *Microscope, 28,* 1980.

8. W. J. Campbell, R. L. Blake, L. L. Brown, E. E. Cather, and J. J. Sjoberg. *Selected Silicate Minerals and Their Asbestiform Varieties: Mineralogical Definitions and Identification-Characterization,* U.S. Bureau of Mines Information Circular 8751, 1977.

9. Walter C. McCrone, *Asbestos Particle Atlas,* Ann Arbor: Ann Arbor Science Publishers, June 1980.

SECTION 2. X-RAY POWDER DIFFRACTION

2.1 Principle and Applicability

The principle of X-ray powder diffraction (XRD) analysis is well established.[1,2] Any solid, crystalline material will diffract an impingent beam of parallel, monochromatic X-rays whenever Bragg's Law,

$\lambda = 2d \sin \theta$,

is satisfied for a particular set of planes in the crystal lattice, where

λ = the X-ray wavelength, Å;
d = the interplanar spacing of the set of reflecting lattice planes, Å; and
θ = the angle of incidence between the X-ray beam and the reflecting lattice planes.

By appropriate orientation of a sample relative to the incident X-ray beam, a diffraction pattern can be generated that, in most cases, will be uniquely characteristic of both the chemical composition and structure of the crystalline phases present.

Unlike optical methods of analysis, however, XRD cannot determine crystal morphology. Therefore, in asbestos analysis, XRD does not distinguish between fibrous and nonfibrous forms of the serpentine and amphibole minerals (Table 2-1). However, when used in conjunction with optical methods such as polarized light microscopy (PLM), XRD techniques can provide a reliable analytical method for the identification and characterization of asbestiform minerals in bulk materials.

For *qualitative analysis* by XRD methods, samples are initially scanned over limited diagnostic peak regions for the serpentine (~7.4 Å) and amphibole (8.2–8.5 Å) minerals (Table 2-2). Standard slow-scanning methods for bulk sample analysis may be used for materials shown by PLM to contain significant amounts of asbestos (>5–10 percent). Detection of minor or trace amounts of asbestos may require special sample preparation and step-scanning analysis. All samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals are submitted to a full (5°–60° 2θ; 1° 2θ/min) qualitative XRD scan, and their diffraction patterns are compared with standard reference powder diffraction patterns[3] to verify initial peak assignments and to identify possible matrix interferences when subsequent quantitative analysis will be performed.

TABLE 2–1—THE ASBESTOS MINERALS AND THEIR NONASBESTIFORM ANALOGS

Asbestiform	Nonasbestiform
SERPENTINE	
Chrysotile	Antigorite, lizardite
AMPHIBOLE	
Anthophyllite asbestos	Anthophyllite
Cummingtonite-grunerite asbestos ("Amosite")	Cummingtonite-grunerite
Crocidolite	Riebeckite
Tremolite asbestos	Tremolite
Actinolite asbestos	Actinolite

TABLE 2–2—PRINCIPAL LATTICE SPACINGS OF ASBESTIFORM MINERALS[a]

Minerals	Principal d-spacings (Å) and relative intensities			JCPDS Powder diffraction file[3] number
Chrysotile	7.37_{100}	3.65_{70}	4.57_{50}	21–543[b]
	7.36_{100} ..	3.66_{80}	2.45_{65}	25–645
	7.10_{100} ..	2.33_{80}	3.55_{70}	22–1162 (theoretical)
"Amosite"	8.33_{100}	3.06_{70}	2.756_{70}	17–745 (nonfibrous)
	8.22_{100} ..	3.060_{85}	3.25_{70}	27–1170 (UICC)
Anthophyllite	3.05_{100}	3.24_{60}	8.26_{55}	9–455
	3.06_{100} ..	8.33_{70}	3.23_{50}	16–401 (synthetic)
Anthophyllite	2.72_{100}	2.54_{100}	3.480_{80}	25–157
Crocidolite	8.35_{100}	3.10_{55}	2.720_{45}	27–1415 (UICC)
Tremolite	8.38_{100}	3.12_{100}	2.705_{90}	13–437[b]
	2.706_{100}	3.14_{95}	8.43_{40}	20–1310[b] (synthetic)
	3.13_{100} ..	2.706_{60}	8.44_{40}	23–666 (synthetic mixture with richterite)

[a] This information is intended as a guide, only. Complete powder diffraction data, including mineral type and source, should be referred to, to ensure comparability of sample and reference materials where possible. Additional precision XRD data on amosite, crocidolite, tremolite, and chrysotile are available from the U.S. Bureaus of Mines.[4]
[b] Fibrosity questionable.

Accurate *quantitative analysis* of asbestos in bulk samples by XRD is critically dependent on particle size distribution, crystallite size, preferred orientation and matrix absorption effects, and comparability of standard reference and sample materials. The most intense diffraction peak that has been shown to be free from interference by prior qualitative XRD analysis is selected for quantitation of each asbestiform mineral. A "thin-layer" method of analysis[5,6] is recommended in which, subsequent to comminution of the bulk material to ~10 μm by suitable cryogenic milling techniques, an accurately known amount of the sample is deposited on a silver membrane filter. The mass of

asbestiform material is determined by measuring the integrated area of the selected diffraction peak using a step-scanning mode, correcting for matrix absorption effects, and comparing with suitable calibration standards. Alternative "thick-layer" or bulk methods,[7 8] may be used for *semiquantitative analysis.*

This XRD method is applicable as a confirmatory method for identification and quantitation of asbestos in bulk material samples that have undergone prior analysis by PLM or other optical methods.

2.2 Range and Sensitivity

The range of the method has not been determined.

The sensitivity of the method has not been determined. It will be variable and dependent upon many factors, including matrix effects (absoprtion and interferences), diagnostic reflections selected, and their relative intensities.

2.3 Limitations

2.3.1 *Interferences*

Since the fibrous and nonfibrous forms of the serpentine and amphibole minerals (Table 2-1) are indistinguishable by XRD techniques unless special sample preparation techniques and instrumentation are used,[9] the presence of nonasbestiform serpentines and amphiboles in a sample will pose severe interference problems in the identification and quantitative analysis of their asbestiform analogs.

The use of XRD for identification and quantitation of asbestiform minerals in bulk samples may also be limited by the presence of other interfering materials in the sample. For naturally occurring materials the commonly associated asbestos-related mineral interferences can usually be anticipated. However, for fabricated materials the nature of the interferences may vary greatly (Table 2-3) and present more serious problems in identification and quantitation.[10] Potential interferences are summarized in Table 2-4 and include the following:

- *Chlorite* has major peaks at 7.19 Å and 3.58 Å That interfere with both the primary (7.36 Å) and secondary (3.66 Å) peaks for chrysotile. Resolution of the primary peak to give good quantitative results may be possible when a step-scanning mode of operation is employed.
- *Halloysite* has a peak at 3.63 Å that interferes with the secondary (3.66 Å) peak for chrysotile.
- *Kaolinite* has a major peak at 7.15 Å that may interfere with the primary peak of chrysotile at 7.36 Å when present at concentrations of >10 percent. However, the secondary chrysotile peak at 3.66 Å may be used for quantitation.
- *Gypsum* has a major peak at 7.5 Å that overlaps the 7.36 Å peak of chrysotile when present as a major sample constituent. This may be removed by careful washing with distilled water, or be heating to 300 °C to convert gypsum to plaster of paris.
- *Cellulose* has a broad peak that partially overlaps the secondary (3.66 Å) chrysotile peak.[8]
- Overlap of major diagnostic peaks of the amphibole asbestos minerals, amosite, anthophyllite, crocidolite, and tremolite, at approximately 8.3 Å and 3.1 Å causes mutual interference when these minerals occur in the presence of one another. In some instances, adquate resolution may be attained by using step-scanning methods and/or by decreasing the collimator slit width at the X-ray port.

TABLE 2-3—COMMON CONSTITUENTS IN INSULATION AND WALL MATERIALS

A. Insulation materials
Chrysotile
"Amosite"
Crocidolite
*Rock wool
*Slag wool
*Fiber glass
Gypsum ($CaSO_4 \cdot 2H_2O$)
Vermiculite (micas)
*Perlite
Clays (kaolin)
*Wood pulp
*Paper fibers (talc, clay, carbonate fillers)
Calcium silicates (synthetic)
Opaques (chromite, magnetite inclusions in serpentine)
Hematite (inclusions in "amosite")
Magnesite
*Diatomaceous earth

B. Spray finishes or paints
Bassanite
Carbonate minerals (calcite, dolomite, vaterite)
Talc
Tremolite
Anthophyllite
Serpentine (including chrysotile)
Amosite
Crocidolite
*Mineral wool
*Rock wool
*Slag wool
*Fiber glass
Clays (kaolin)
Micas
Chlorite
Gypsum ($CaSO_4 \cdot 2H_2O$)
Quartz
*Organic binders and thickeners
Hyrdomagnesite
Wollastonite
Opaques (chromite, magnetite inclusions in serpentine)
Hematite (inclusions in "amosite")

*Amorphous materials___contribute only to overall scattered radiation and increased background radiation.

TABLE 2–4—INTERFERENCES IN XRD ANALYSIS ASBESTIFORM MINERALS

Asbestiform mineral	Primary diagnostic peaks (approximate d-spacings, in Å)	Interference
Serpentine		
Chrysotile	7.4	Nonasbestiform serpentines (antigorite, lizardite)
		Chlorite
		Kaolinite
		Gypsum
	3.7	Chlorite
		Halloysite
		Cellulose
Amphibole		
"Amosite" Anthophyllite Crocidolite Tremolite	3.1	Nonasbestiform amphiboles (cummingtonite-grunerite, anthophyllite, riebeckite, tremolite)
		Mutual interferences
		Carbonates
		Talc
	8.3	Mutual interferences

- *Carbonates* may also interfere with quantitative analysis of the amphibole asbestos minerals, amosite, anthophyllite, crocidolite, and tremolite. Calcium carbonate ($CaCO_3$) has a peak at 3.035 Å that overlaps major amphibole peaks at approximately 3.1 Å when present in concentrations of >5 percent. Removal of carbonates with a dilute acid wash is possible; however, if present, chrysotile may be partially dissolved by this treatment.[11]
- A major *talc* peak at 3.12 Å interferes with the primary tremolite peak at this same position and with secondary peaks of crocidolite (3.10 Å), amosite (3.06 Å), and anthophyllite (3.05 Å). In the presence of talc, the major diagnostic peak at approximately 8.3 Å should be used for quantitation of these asbestiform minerals.

The problem of intraspecies and matrix interferences is further aggravated by the variability of the silicate mineral powder diffraction patterns themselves, which often makes definitive identification of the asbestos minerals by comparison with standard reference diffraction patterns difficult. This variability results from alterations in the crystal lattice associated with differences in isomorphous substitution and degree of crystallinity. This is especially true for the amphiboles. These minerals exhibit a wide variety of very similar chemical compositions, with the result being that their diffraction patterns are chracterized by having major (110) reflections of the monoclinic amphiboles and (210) reflections of the orthorhombic anthophyllite separated by less than 0.2 Å.[12]

2.3.2 *Matrix Effects*

If a copper X-ray source is used, the presence of iron at high concentrations in a sample will result in significant X-ray fluorescence, leading to loss of peak intensity along with increased background intensity and an overall decrease in sensitivity. This situation may be corrected by choosing an X-ray source other than copper; however, this is often accompanied both by loss of intensity and by decreased resolution of closely spaced reflections. Alternatively, use of a diffracted beam monochromator will reduce background fluorescent raditation, enabling weaker diffraction peaks to be detected.

X-ray absorption by the sample matrix will result in overall attenuation of the diffracted beam and may seriously interfere with quantitative analysis. Absorption effects may be minimized by using sufficiently "thin" samples for analysis.[5 13 14] However, unless absorption effects are known to be the same for both samples and standards, appropriate corrections should be made by referencing diagnostic peak areas to an internal standard[7 8] or filter substrate (Ag) peak.[5 6]

2.3.3 *Particle Size Dependence*

Because the intensity of diffracted X-radiation is particle-size dependent, it is essential for accurate quantitative analysis that both sample and standard reference materials have similar particle size distributions. The optimum particle size range for quantitative analysis of asbestos by XRD has been reported to be 1 to 10 μ m.[15] Comparability of sample and standard reference material particle size distributions should be verified by optical microscopy (or another suitable method) prior to analysis.

2.3.4 *Preferred Orientation Effects*

Preferred orientation of asbestiform minerals during sample preparation often poses a serious problem in quantitative analysis by XRD. A number of techniques have been developed for reducing preferred orientation effects in "thick layer" samples.[7 8 15] However, for "thin" samples on membrane filters, the preferred orientation effects seem to be both reproducible and favorable to enhancement of the principal diagnostic reflections of asbestos minerals, actually increasing the overall sensitivity of the method[12 14] (Further investigation into preferred orientation effects in both thin layer and bulk samples is required.)

2.3.5 *Lack of Suitably Characterized Standard Materials*

The problem of obtaining and characterizing suitable reference materials for asbestos analysis is clearly recognized. NIOSH has recently directed a major research effort toward the preparation and characterization of analytical reference materials, including asbestos standards;[16 17] however, these are not available in large quantities for routine analysis.

In addition, the problem of ensuring the comparability of standard reference and sample materials, particularly regarding crystallite size, particle size distribution, and degree of crystallinity, has yet to be adequately addressed. For example, Langer et al.[18] have observed that in insulating matrices, chrysotile tends to break open into bundles more frequently than amphiboles. This results in a line-broadening effect with a resultant decrease in sensitivity. Unless this effect is the same for both standard and sample materials, the amount of chrysotile in the sample will be underestimated by XRD analysis. To minimize this problem, it is recommended that standardized matrix reduction procedures be used for both sample and standard materials.

2.4 Precision and Accuracy

Precision of the method has not been determined.

Accuracy of the method has not been determined.

2.5 Apparatus

2.5.1 *Sample Preparation*

Sample preparation apparatus requirements will depend upon the sample type under consideration and the kind of XRD analysis to be performed.

- *Mortar and Pestle:* Agate or porcelain.
- *Razor Blades*
- *Sample Mill:* SPEX, Inc., freezer mill or equivalent.
- *Bulk Sample Holders*
- *Silver Membrane Filters:* 25-mm diameter, 0.45-μ m pore size. Selas Corp. of America, Flotronics Div., 1957 Pioneer Road, Huntington Valley, PA 19006.
- *Microscope Slides*
- *Vacuum Filtration Apparatus:* Gelman No. 1107 or equivalent, and side-arm vacuum flask.
- *Microbalance*
- *Ultrasonic Bath or Probe:* Model W140, Ultrasonics, Inc., operated at a power density of approximately 0.1 W/mL, or equivalent.
- *Volumetric Flasks:* 1-L volume.
- *Assorted Pipettes*
- *Pipette Bulb*
- *Nonserrated Forceps*
- *Polyethylene Wash Bottle*
- *Pyrex Beakers:* 50-mL volume.
- *Desiccator*
- *Filter Storage Cassettes*
- *Magnetic Stirring Plate and Bars*
- *Porcelain Crucibles*
- *Muffle Furnace or Low Temperature Asher*

2.5.2 *Sample Analysis*

Sample analysis requirements include an X-ray diffraction unit, equipped with:

- *Constant Potential Generator; Voltage and mA Stabilizers*
- *Automated Diffractometer with Step-Scanning Mode*
- *Copper Target X-Ray Tube:* High intensity, fine focus, preferably.
- *X-Ray Pulse Height Selector*
- *X-Ray Detector* (with high voltage power supply): Scintillation or proportional counter.
- *Focusing Graphite Crystal Monochromator;* or *Nickel Filter* (if copper source is used, and iron fluorescence is not a serious problem).
- *Data Output Accessories:*
  - *Strip Chart Recorder*
  - *Decade Scaler/Timer*
  - *Digital Printer*
- *Sample Spinner* (optional).
- *Instrument Calibration Reference Specimen:* α-quartz reference crystal (Arkansas quartz standard, #180-147-00, Philips Electronics Instruments, Inc., 85 McKee Drive, Mahwah, NJ 07430) or equivalent.

2.6 Reagents

2.6.1 *Standard Reference Materials*

The reference materials listed below are intended to serve as a guide. Every attempt should be made to acquire pure reference materials that are comparable to sample materials being analyzed.

- *Chrysotile:* UICC Canadian, or NIEHS Plastibest. (UICC reference materials available from: UICC, MRC Pneumoconiosis Unit, Llandough Hospital, Penarth, Glamorgan, CF61XW, UK).
- *Crocidolite:* UICC
- *Amosite:* UICC
- *Anthophyllite:* UICC
- *Tremolite Asbestos:* Wards Natural Science Establishment, Rochester, N.Y.: Cyprus Research Standard, Cyprus Research, 2435 Military Ave., Los Angeles, CA 90064 (washed with dilute HCl to remove small amount of calcite impurity); India tremolite, Rajasthan State, India.
- *Actinolite Asbestos*

2.6.2 *Adhesive*

Tape, petroleum jelly, etc. (for attaching silver membrane filters to sample holders).

2.6.3 *Surfactant*

1 percent aerosol OT aqueous solution or equivalent.

2.6.4 *Isopropanol*

ACS Reagent Grade.

2.7 Procedure

2.7.1 *Sampling*

Samples for analysis of asbestos content shall be collected as specified in EPA Guidance Document #C0090, *Asbestos-Containing Materials in School Buildings.*[10]

2.7.2 *Analysis*

All samples must be analyzed initially for asbestos content by PLM. XRD should be used as an auxiliary method when a second, independent analysis is requested.

NOTE: Asbestos is a toxic substance. All handling of dry materials should be performed in an operating fume hood.

2.7.2.1 *Sample Preparation*

The method of sample preparation required for XRD analysis will depend on: (1). The condition of the sample received (sample size, homogeneity, particle size distribution, and overall composition as determined by PLM); and (2) the type of XRD analysis to be performed (qualitative, quantitative, thin layer or bulk).

Bulk materials are usually received as inhomogeneous mixtures of complex composition with very wide particle size distributions. Preparation of a homogeneous, representative sample from asbestos-containing materials is particularly difficult because the fibrous nature of the asbestos minerals inhibits mechanical mixing and stirring, and because milling procedures may cause adverse lattice alterations.

A discussion of specific matrix reduction procedures is given below. Complete methods of sample preparation are detailed in Sections 2.7.2.2 and 2.7.2.3.

NOTE: All samples should be examined microscopically before and after each matrix reduction step to monitor changes in sample particle size, composition, and crystallinity, and to ensure sample representativeness and homogeneity for analysis.

2.7.2.1.1 *Milling*— Mechanical milling of asbestos materials has been shown to decrease fiber crystallinity, with a resultant decrease in diffraction intensity of the specimen; the degree of lattice alteration is related to the duration and type of milling process.[19, 20, 21, 22] Therefore, all milling times should be kept to a minimum.

For *qualitative analysis,* particle size is not usually of critical importance and initial characterization of the material with a minimum of matrix reduction is often desirable to document the composition of the sample as received. Bulk samples of very large particle size (>2-3 mm) should be comminuted to ~100 μm. A mortar and pestle can sometimes be used in size reduction of soft or loosely bound materials though this may cause matting of some samples. Such samples may be reduced by cutting with a razor blade in a mortar, or by grinding in a suitable mill (e.g., a microhammer mill or equivalent). When using a mortar for grinding or cutting, the sample should be moistened with ethanol, or some other suitable wetting agent, to minimize exposures.

For accurate, reproducible *quantitative analysis,* the particle size of both sample and standard materials should be reduced to ~10 μm (see Section 2.3.3). Dry ball milling at liquid nitrogen temperatures (e.g., Spex Freezer Mill, or equivalent) for a maximum time of 10 min. is recommended to obtain satisfactory particle size distributions while protecting the integrity of the crystal lattice.[5] Bulk samples of very large particle size may require grinding in two stages for full matrix reduction to <10 μm.[8, 16]

Final particle size distributions should always be verified by optical microscopy or another suitable method.

2.7.2.1.2 *Low temperature ashing*— For materials shown by PLM to contain large amounts of gypsum, cellulosic, or other organic materials, it may be desirable to ash the samples prior to analysis to reduce background radiation or matrix interference. Since chrysotile undergoes dehydroxylation at temperatures between 550 °C and and 650 °C, with subsequent transformation to forsterite,[23, 24] ashing temperatures should be kept below 500 °C. Use of a low temperature asher is recommended. In all cases, calibration of the oven is essential to ensure that a maximum ashing temperature of 500 °C is not exceeded.

2.7.2.1.3 *Acid leaching*—Because of the interference caused by gypsum and some carbonates in the detection of asbestiform minerals by XRD (see Section 2.3.1), it may be necessary to remove these interferents by a simple acid leaching procedure prior to analysis (see Section 1.7.2.2).

2.7.2.2 *Qualitative Analysis*

2.7.2.2.1 *Initial screening of bulk material*— Qualitative analysis should be performed on a representative, homogeneous portion of the sample with a minimum of sample treatment.

1. Grind and mix the sample with a mortar and pestle (or equivalent method, see Section 2.7.2.1.1) to a final particle size sufficiently small (~100 μm) to allow adequate packing into the sample holder.

2. Pack the sample into a standard bulk sample holder. Care should be taken to ensure that a representative portion of the

milled sample is selected for analysis. Particular care should be taken to avoid possible size segregation of the sample. (Note: Use of a back-packing method[25] of bulk sample preparation may reduce preferred orientation effects.)

3. Mount the sample on the diffractometer and scan over the diagnostic peak regions for the serpentine (~67.4 Å) and amphibole (8.2–8.5 Å) minerals (see Table 2–2). The X-ray diffraction equipment should be optimized for intensity. A slow scanning speed of 1° 2θ/min is recommended for adequate resolution. Use of a sample spinner is recommended.

4. Submit all samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals to a full qualitative XRD scan (5°–60° 2θ; 1°2θ/min) to verify initial peak assignments and to identify potential matrix interferences when subsequent quantitative analysis is to be performed.

5. Compare the sample XRD pattern with standard reference powder diffraction patterns (i.e., JCPDS powder diffraction data[3] or those of other well-characterized reference materials). Principal lattice spacings of asbestiform minerals are given in Table 2–2; common constituents of bulk insulation and wall materials are listed in Table 2–3.

2.7.2.2.2 *Detection of minor or trace constituents*— Routine screening of bulk materials by XRD may fail to detect small concentrations (<5 percent) of asbestos. The limits of detection will, in general, be improved if matrix absorption effects are minimized, and if the sample particle size is reduced to the optimal 1 to 10 μm range, provided that the crystal lattice is not degraded in the milling process. Therefore, in those instances where confirmation of the presence of an asbestiform mineral at very low levels is required, or where a negative result from initial screening of the bulk material by XRD (see Section 2.7.2.2.1) is in conflict with previous PLM results, it may be desirable to prepare the sample as described for quantitative analysis (see Section 2.7.2.3) and step-scan over appropriate 2θ ranges of selected diagnostic peaks (Table 2–2). Accurate transfer of the sample to the silver membrane filter is not necessary unless subsequent quantitative analysis is to be performed.

2.7.2.3 *Quantitative Analysis*

The proposed method for quantitation of asbestos in bulk samples is a modification of the NIOSH-recommended thin-layer method for chrysotile in air.[5] A thick-layer or bulk method involving pelletizing the sample may be used for semiquantitative analysis;[7,8] however, this method requires the addition of an internal standard, use of a specially fabricated sample press, and relatively large amounts of standard reference materials. Additional research is required to evaluate the comparability of thin- and thick-layer methods for quantitative asbestos analysis.

For quantitative analysis by thin-layer methods, the following procedure is recommended:

1. Mill and size all or a substantial representative portion of the sample as outlined in Section 2.7.2.1.1.

2. Dry at 100 °C for 2 hr; cool in a desiccator.

3. Weigh accurately to the nearest 0.01 mg.

4. Samples shown by PLM to contain large amounts of cellulosic or other organic materials, gypsum, or carbonates, should be submitted to appropriate matrix reduction procedures described in Sections 2.7.2.1.2 and 2.7.2.1.3. After ashing and/or acid treatment, repeat the drying and weighing procedures described above, and determine the percent weight loss; L.

5. Quantitatively transfer an accurately weighed amount (50–100 mg) of the sample to a 1-L volumetric flask with approximately 200 mL isopropanol to which 3 to 4 drops of surfactant have been added.

6. Ultrasonicate for 10 min at a power density of approximately 0.1 W/mL, to disperse the sample material.

7. Dilute to volume with isopropanol.

8. Place flask on a magnetic stirring plate. Stir.

9. Place a silver membrane filter on the filtration apparatus, apply a vacuum, and attach the reservoir. Release the vacuum and add several milliliters of isopropanol to the reservoir. Vigorously hand shake the asbestos suspension and immediately withdraw an aliquot from the center of the suspension so that total sample weight, W_T, on the filter will be approximately 1 mg. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and resume the procedure with a clean pipet. Transfer the aliquot to the reservoir. Filter rapidly under vacuum. Do not wash the reservoir walls. Leave the filter apparatus under vacuum until dry. Remove the reservoir, release the vacuum, and remove the filter with forceps. (Note: Water-soluble matrix interferences such as gypsum may be removed at this time by careful washing of the filtrate with distilled water. Extreme care should be taken not to disturb the sample.)

10. Attach the filter to a flat holder with a suitable adhesive and place on the diffractometer. Use of a sample spinner is recommended.

11. For each asbestos mineral to be quantitated select a reflection (or reflections) that has been shown to be free from interferences by prior PLM or qualitative XRD analysis and that can be used unambiguously as an index of the amount of material present in the sample (see Table 2–2).

12. Analyze the selected diagnostic reflection(s) by step scanning in increments of

0.02° 2θ for an appropriate fixed time and integrating the counts. (A fixed count scan may be used alternatively; however, the method chosen should be used consistently for all samples and standards.) An appropriate scanning interval should be selected for each peak, and background corrections made. For a fixed time scan, measure the background on each side of the peak for one-half the peak-scanning time. The net intensity, I_a, is the difference between the peak integrated count and the total background count.

13. Determine the net count, I_{Ag}, of the filter 2.36 Å silver peak following the procedure in step 12. Remove the filter from the holder, reverse it, and reattach it to the holder. Determine the net count for the unattenuated silver peak, $I_{\mathring{A}g}$. Scan times may be *less* for measurement of silver peaks than for sample peaks; however, they should be *constant* throughout the analysis.

14. Normalize all raw, net intensities (to correct for instrument instabilities) by referencing them to an external standard (e.g., the 3.34 Å peak of an α-quartz reference crystal). After each unknown is scanned, determine the net count, I_r, of the reference specimen following the procedure in step 12. Determine the normalized intensities by dividing the peak intensities by I_r.

$$\hat{I}_a = \frac{I_a}{I_r^o}, \quad \hat{I}_{Ag} = \frac{I_{Ag}}{I_r^o}, \quad \text{and} \quad \hat{I}_{Ag}^o = \frac{I_{Ag}^o}{I_r^o}$$

2.8 Calibration

2.8.1 *Preparation of Calibration Standards*

1. Mill and size standard asbestos materials according to the procedure outlined in Section 2.7.2.1.1. *Equivalent, standardized matrix reduction and sizing techniques should be used for both standard and sample materials.*

2. Dry at 100 °C for 2 hr; cool in a desiccator.

3. Prepare two suspensions of each standard in isopropanol by weighing approximately 10 and 50 mg of the dry material to the nearest 0.01 mg. Quantitatively transfer each to a 1-L volumetric flask with approximately 200 mL isopropanol to which a few drops of surfactant have been added.

4. Ultrasonicate for 10 min at a power density of approximately 0.1 W/mL, to disperse the asbestos material.

5. Dilute to volume with isopropanol.

6. Place the flask on a magnetic stirring plate. Stir.

7. Prepare, in triplicate, a series of at least five standard filters to cover the desired analytical range, using appropriate aliquots of the 10 and 50 mg/L suspensions and the following procedure.

Mount a silver membrane filter on the filtration apparatus. Place a few milliliters of isopropanol in the reservoir. Vigorously hand shake the asbestos suspension and immediately withdraw an aliquot from the center of the suspension. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and resume the procedure with a clean pipet. Transfer the aliquot to the reservoir. Keep the tip of the pipet near the surface of the isopropanol. Filter rapidly under vacuum. Do not wash the sides of the reservoir. Leave the vacuum on for a time sufficient to dry the filter. Release the vacuum and remove the filter with forceps.

2.8.2 *Analysis of Calibration Standards*

1. Mount each filter on a flat holder. Perform step scans on selected diagnostic reflections of the standards and reference specimen using the procedure outlined in Section 2.7.2.3, step 12, and the same conditions as those used for the samples.

2. Determine the normalized intensity for each peak measured, $\hat{I}_{std}$, as outlined in Section 2.7.2.3, step 14.

2.9 Calculations

For each asbestos reference material, calculate the exact weight deposited on each standard filter from the concentrations of the standard suspensions and aliquot volumes. Record the weight, w, of each standard. Prepare a calibration curve by regressing $\hat{I}2_{std}$ on w. Poor reproducibility (±15 percent RSD) at any given level indicates problems in the sample preparation technique, and a need for new standards. The data should fit a straight line equation.

Determine the slope, m, of the calibration curve in counts/microgram. The intercept, b, of the line with the $\hat{I}_{std}$ axis should be approximately zero. A large negative intercept indicates an error in determining the background. This may arise from incorrectly measuring the baseline or from interference by another phase at the angle of background measurement. A large positive intercept indicates an error in determining the baseline or that an impurity is included in the measured peak.

Using the normalized intensity, $\hat{I}_{Ag}$, for the attenuated silver peak of a sample, and the corresponding normalized intensity from the unattenuated silver peak, $\hat{I}_{\mathring{A}g}$, of the sample filter, calculate the transmittance, T, for each sample as follows:[26 27]

$$T = \frac{\hat{I}_{Ag}}{I^{\circ}_{Ag}}$$

Determine the correction factor, f(T), for each sample according to the formula:

$$f(T) = \frac{-R\ (\ln T)}{1-T^{R}}$$

where

$$R = \frac{\sin \Theta_{Ag}}{\sin \Theta_{a}}$$

θ_{Ag}=angular position of the measured silver peak (from Bragg's Law), and

θ_a=angular position of the diagnostic asbestos peak.

Calculate the weight, W_a, in micrograms, of the asbestos material analyzed for in each sample, using the appropriate calibration data and absorption corrections:

$$W_a = \frac{\hat{I}_a f(t) - b}{m}$$

Calculate the percent composition, P_a, of each asbestos mineral analyzed for in the parent material, from the total sample weight, W_T, on the filter:

$$P_a = \frac{W_a(1-.01L)}{W_T} \times 100$$

where

P_a=percent asbestos mineral in parent material;

W_a=mass of asbestos mineral on filter, in µg;

W_T=total sample weight on filter, in µg;

L=percent weight loss of parent material on ashing and/or acid treatment (see Section 2.7.2.3).

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Subpart F [Reserved]

Subpart G—Asbestos Worker Protection

SOURCE: 65 FR 69216, Nov. 15, 2000, unless otherwise noted.

§ 763.120 What is the purpose of this subpart?

This subpart protects certain State and local government employees who are not protected by the Asbestos Standards of the Occupational Safety and Health Administration (OSHA). This subpart applies the OSHA Asbestos Standards in 29 CFR 1910.1001 and 29 CFR 1926.1101 to these employees.

§ 763.121 Does this subpart apply to me?

If you are a State or local government employer and you are not subject to a State asbestos standard that OSHA has approved under section 18 of the Occupational Safety and Health Act or a State asbestos plan that EPA has exempted from the requirements of this subpart under § 763.123, you must follow the requirements of this subpart to protect your employees from occupational exposure to asbestos.

§ 763.122 What does this subpart require me to do?

If you are a State or local government employer whose employees perform:

(a) Construction activities identified in 29 CFR 1926.1101(a), you must:

(1) Comply with the OSHA standards in 29 CFR 1926.1101.

(2) Submit notifications required for alternative control methods to the Director, National Program Chemicals Division (7404), Office of Pollution Prevention and Toxics, Environmental Protection Agency, 1200 Pennsylvania Ave., NW., Washington, DC 20460.

(b) Custodial activities not associated with the construction activities identified in 29 CFR 1926.1101(a), you must comply with the OSHA standards in 29 CFR 1910.1001.

(c) Repair, cleaning, or replacement of asbestos-containing clutch plates and brake pads, shoes, and linings, or removal of asbestos-containing residue from brake drums or clutch housings, you must comply with the OSHA standards in 29 CFR 1910.1001.

§ 763.123 May a State implement its own asbestos worker protection plan?

This section describes the process under which a State may be exempted from the requirements of this subpart.

(a) *States seeking an exemption.* If your State wishes to implement its own asbestos worker protection plan, rather than complying with the requirements of this subpart, your State must apply

DRAFT

MODIFIED ELUTRIATOR METHOD FOR THE DETERMINATION OF ASBESTOS IN SOILS AND BULK MATERIAL

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TABLE OF CONTENTS

TABLE OF CONTENTS (CONT.)

1.0 INTRODUCTION

This elutriator method is a sampling and analysis method for the determination of asbestos in soils and bulk materials. It is specifically designed to provide measurements that can be related to exposure and risk.

Measurements derived using this method can be combined with published dust emission and dispersion models to provide predictions of airborne asbestos concentrations that may result when asbestos-containing bulk materials are disturbed by human activities or natural phenomena. The resulting predictions are sufficiently accurate to support risk assessment and risk management (see, for example, Berman 2000). Moreover, the distribution of the sizes and shapes of the asbestos structures that are released when bulk materials are disturbed is largely preserved by the procedures described in this method for sample collection, handling, preparation, and analysis. Therefore, concentrations reported using this method preserve the size and shape information that is required to assess the risks associated with predicted exposures (see Berman and Crump 1999a).

The elutriator method documented in this report provides estimates of the concentration of asbestos structures that satisfy the dimensional constraints of a particular exposure index, which is the specific index defined in a companion protocol for conducting asbestos risk assessments (Berman and Crump 1999b). An exposure index is a concisely defined set of structure sizes, shapes, and mineralogy that represent the range of asbestos structures that are biologically active and therefore contribute to risk.

> Note, even if there is a desire to evaluate asbestos-related risks based on theories that rely on exposure indices that differ from the one defined herein, this method can still be used to provide the measurements required to predict exposure and risk. The only change needed would be to modify the counting rules (presented in Section 10.1.1), which define the sizes, shapes, and mineralogy of asbestos structures to be included in the determination of asbestos concentrations.

This method was adapted from the "Superfund Method for the Determination of Releasable Asbestos in Soils and Bulk Materials" (Berman and Kolk 1997) and incorporates several cost-saving modifications and other refinements. In both methods, samples are collected in a manner suitable for providing representative measurements of the concentration of asbestos in the matrix sampled, prepared using a dust generator, and analyzed by transmission electron microscopy (TEM). Modifications incorporated into this method include changes to the design and operation of the required dust generator, changes in the manner in which asbestos filters that are collected in the dust generator are prepared for TEM analysis, changes in the counting

rules defined for conducting the analysis, and changes in the manner in which the resulting concentrations are calculated and reported. Specifications for the required modifications to the dust generator are also provided. Design specifications for the original dust generator are provided in the Superfund Method (Berman and Kolk 1997).

Procedures defined for sample collection and field preparation in this method yield 40 g to 70 g samples of bulk materials that are unmodified other than limited coarse crushing (for consolidated materials) and coarse sieving to separate and remove particles larger than 1 cm (3/8ths in.) in diameter. These samples are typically (but not necessarily) derived by sub-sampling a homogenized composite of multiple, large-volume (approximately 1 kg) samples that are collected from an array designed to provide a representative sample of the bulk matrix of interest. The final 40 g to 70 g sub-samples are then sent to the laboratory for analysis.

> Note: smaller volume (mass) samples can also be analyzed by this method. It is simply recommended that samples smaller than approximately 40 g be diluted with a measured mass of washed, play sand (Section 8.3) prior to analysis.

Samples received by the laboratory are placed in the tumbler of a dust generator, which is designed to entrain the fine fraction of the sample in an air stream. The air stream is then passed through the vertical elutriator of the dust generator, which concentrates the asbestos by removing everything except the respirable fraction of fines from the sample. The resulting respirable fraction (which includes the respirable asbestos structures) is then deposited on a filter. The filter is weighed to determine the mass of respirable dust that is deposited on the filter and grid specimens are prepared for TEM analysis from selected portions of the filter using a direct transfer technique. An optional, indirect transfer technique is also described. Results are then reported as the ratio of the number of asbestos structures (within the size range of interest) per unit mass of respirable dust. This is precisely the manner in which asbestos concentrations need to be reported when they are used as inputs to published dust models for predicting airborne asbestos concentrations (Berman 2000).

As reported in this document, this method focuses on requirements for the collection, preparation, and analysis of single samples that are obtained from the field. During a site investigation (as previously indicated), samples will typically be collected from multiple locations that are arranged in an array designed to provide measurements suitable for deriving a representative (i.e. unbiased) estimate of the concentration of asbestos over the sampled matrix as a whole. Thus, proper design of a comprehensive sampling strategy, which includes a detailed design for the array of sampling locations, is also critical to the success of an investigation. However, design of a sampling strategy is necessarily site specific and site-specific considerations are beyond the scope of this document. For further guidance on developing appropriate sampling strategies, see Berman and Chesson (unpublished).

The sensitivity and precision of this method are now well defined (see Sections 2.4 and 2.5). Although the validity of the overall approach for predicting exposure has only been demonstrated in a single study (Berman 2000) heretofore, it is evident from that study that the limitations to the accuracy of such predictions lie primarily with the emission and dispersion models that need to be coupled with analytical results from this method. Nevertheless, a limited number of additional validation studies (to test the accuracy of predictions over a broader range of bulk matrices and release scenarios) would serve to better validate this method.

2.0 BACKGROUND

As indicated previously, this method was specifically developed to provide measurements suitable for supporting risk assessment. To support risk assessment, the method satisfies certain requirements including the need to:

- provide concentration measurements for the specific set of asbestos structure sizes and shapes that contribute to adverse biological effects;
- provide bulk concentration measurements reported in units that are appropriate for inputs to emission and transport models, which can be used to predict airborne exposure concentrations associated with release and dispersion scenarios of interest;
- achieve sufficient accuracy to support adequately accurate exposure (and risk) predictions;
- achieve adequate sensitivity to allow measurement of asbestos over the entire range of concentrations that potentially pose unacceptable risk;
- achieve adequate precision over the range of asbestos concentrations of interest;
- incorporate procedures for sample handling, preparation, and analysis that are insensitive to subjective application so that they are readily reproducible within and between laboratories that may offer the method commercially; and
- readily accommodate a broad range of (natural and anthropogenic) bulk materials for which determining the concentration of asbestos may be of interest.

An additional consideration addressed during the development of this method is the need to control sampling and analysis costs, which is also addressed below.

2.1 BIOLOGICALLY RELEVANT ASBESTOS STRUCTURES

Asbestos poses a threat to human health when dust is released and the resulting structures are inhaled. Whether asbestos also poses a threat to human health when it is ingested is less clear. There is no direct human evidence that ingested asbestos causes disease and the existing animal studies are equivocal (U.S. EPA 1986). Therefore, the focus of this method is to provide bulk measurements that can ultimately be used to predict airborne (inhalation) exposures.

There is strong evidence and general agreement within the scientific community that the risk of asbestos-related disease attributable to a particular exposure is a function of dose (measured in terms of the number of inhaled structures). Further, in addition to structure number concentration, both the mineralogy and sizes and shapes of the inhaled structures determine the magnitude of the risk (see, for example, Berman and Crump 1999a). Therefore, bulk measurements that are collected to support asbestos risk assessment must enumerate and distinguish asbestos structures within relevant size categories and identify their mineralogy.

In the recently completed protocol for assessing asbestos risks (Berman and Crump 1999b), a specific exposure index was defined that is expected to capture the range of asbestos structure sizes and shapes that contribute to risk. This exposure index was developed based on an extensive review of the literature and results from supplemental studies. The exposure index is concisely defined as the weighted sum of the concentrations of asbestos structures in two, related size categories:

$$C_{opt} = 0.003C_S + 0.997C_L \qquad (2.1)$$

where:

"C_{opt}" is the concentration of asbestos expressed in terms of the recommended exposure index. Such concentrations are expected to closely relate to risk;

"C_S" is the concentration of asbestos structures between 5 and 10 µm in length that are also thinner than 0.5 µm; and

"C_L" is the concentration of asbestos structures longer than 10 µm that are also thinner than 0.5 µm.

To provide measurements that can be used to derive concentration estimates reported in terms of the above-described exposure index, it is necessary to separately enumerate asbestos structures that are longer than 5 µm and thinner than 0.5 µm, to distinguish those also longer than 10 µm, and to identify the mineralogy of all such structures. Structures that satisfy the dimensional constraints defined above have been termed, "protocol structures." The rules for counting and characterizing asbestos structures during analysis that are incorporated into this method (Section 10.1) are designed specifically for enumerating protocol structures.

> Note, it has also been determined, based on the literature review and supplemental studies (Berman and Crump 1999a), that samples prepared for TEM analysis by a direct transfer technique relate best to risk. Therefore, a direct transfer technique has been incorporated into this method (Section 9.1). However, an optional, indirect transfer technique is also presented (Section 9.2).

This is because analytical results derived from samples prepared by indirect transfer may sometimes correlate with results from paired samples prepared by direct transfer (within specific matrices at specific sites). Due to the ability to optimize loading, such analyses may occasionally prove less expensive than analyses of samples prepared by direct transfer.

2.2 BULK MEASUREMENTS FOR EMISSION AND DISPERSION MODELING

As indicated previously, bulk measurements derived using this method are intended to be combined with published dust emission and dispersion models to predict airborne asbestos concentrations that may result when asbestos-containing bulk materials are disturbed by human activities or natural phenomena. Based on a dimensional analysis of such models (Berman 2000), a basic requirement for bulk measurements used for this purpose is that results be reported in units of the number of asbestos structures per mass of respirable dust (S/g_{PM10})[1]. Therefore, this method incorporates procedures for simultaneous determination of the number of asbestos structures and the respirable particulate mass that are liberated from a sample during dust generation (Section 8.5). and results are reported as the required ratio: S/g_{PM10}.

Experience gained from use of this method indicates that reporting asbestos concentrations as the ratio of asbestos to dust (which is required in this method) offers an additional, substantial advantage over other, more traditional methods for measuring asbestos in bulk materials. This relates to the ability to account for the efficiency with which sample preparation liberates the asbestos from a bulk sample so that it is available (visible) for analysis.

When samples are analyzed chemically, typically, they are first dissolved. Dissolution degrades the sample quantitatively to its component molecules. Thus, the total number of molecules of interest that were present in the original sample are available for detection during analysis. In contrast, bulk asbestos samples must typically be degraded mechanically to liberate asbestos for analysis. Commonly, such mechanical degradation is performed to achieve some pre-defined, but arbitrary size specification. However, the corresponding efficiency with which the asbestos structures in the sample have been liberated so that they are available (visible) is never known. Moreover, mechanical degradation can never be sufficiently comprehensive to assure liberation of all the asbestos structures in a sample, because such aggressive action would unavoidably alter and degrade the asbestos structures themselves. It is thus

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[1] "PM_{10}" is an abbreviation that is commonly used to represent respirable dust, which is defined as particulate matter exhibiting an aerodynamic equivalent diameter less than 10 µm (Raabe 1984). The aerodynamic equivalent diameter of a particle is the diameter of a hypothetical spherical particle of unit density that exhibits the same settling velocity in air as the particle of interest.

impossible to quantitatively liberate asbestos structures from a bulk sample by mechanical means.

When bulk samples are (naturally or mechanically) degraded and liberate asbestos, fines (including respirable dust) are unavoidably produced in the process. Further, the quantity of respirable dust that is produced is a direct function of the degree of such degradation. Thus, measuring the quantity of respirable dust present in a sample provides at least a rough measure of the efficiency with which asbestos may have been liberated from the sample so that it is available for detection during analysis. Therefore, dividing counts of the number of asbestos structures liberated from a sample by the mass of respirable dust that is simultaneously liberated normalizes the reported asbestos concentrations to account for the degree of degradation of the sample achieved during preparation and analysis.

By reporting asbestos concentrations as the ratio of asbestos to dust, this method (and its predecessor the Superfund Method) provides a measure of asbestos that is an inherent property of the material analyzed, much as a chemical concentration is an inherent property of the material analyzed. Such a measure of asbestos concentration is robust (i.e. relatively insensitive) to the manner in which a sample is handled and prepared. This facilitates reproducibility when the method is applied because subjective effects attributable to differences in the way that individuals or laboratories handle or prepare samples should not affect the results of analyses performed using this method. The same cannot be said of other, more traditional methods commonly used for the determination of asbestos in bulk materials.

That reporting measurements as the ratio of asbestos structure number to respirable dust mass normalizes reported concentrations so that they are robust to subjective handling and preparation (as well as natural in-place weathering) is indicated by comparing results from a recent (unpublished) study in which measurements were collected from ground and unground soil samples.

In the recent study, paired splits were prepared from three soil samples known to contain tremolite asbestos. One split of each pair was prepared as recommended in this method (i.e. by sieving to remove particles larger than 1 cm). The split was then weighed and placed directly in the tumbler of the dust generator for conditioning, dust generation, and analysis (Sections 8.5 and 10.1). The second split of each pair was ground so that the entire sample passed through a 200-mesh sieve prior to weighing, conditioning in the tumbler, dust generation, and analysis.

Asbestos concentrations were measured in the above-described sample splits using this method and results were reported for each of two structure size categories: (1) protocol structures and (2) phase contrast microscopy equivalent (PCME) structures. PCME structures are those that are longer than 5 µm, thicker than 0.25 µm,

and exhibit an aspect (length to width) ratio greater than 3:1. Both sets of results are presented in the following table.

Table 2-1:
Asbestos Concentrations Measured in Paired Sample Splits that Were Ground and Unground, Respectively

	Asbestos Protocol Structure Concentrations (S/g_{PM10})	
Sample Number	**from Unground Split**	**from Ground Split**
Sample Number 1	2.64×10^7	1.27×10^7
Sample Number 2	2.17×10^7	1.33×10^7
Sample Number 3	1.38×10^7	1.10×10^7

	Asbestos PCME Structure Concentrations (S/g_{PM10})	
Sample Number	**from Unground Split**	**from Ground Split**
Sample Number 1	6.40×10^7	1.08×10^8
Sample Number 2	1.20×10^8	1.28×10^8
Sample Number 3	1.39×10^8	5.95×10^7

The concentrations reported in Table 2-1 each represent the mean of duplicate (or triplicate) analyses. Each analysis is derived from one of a set of filters collected at differing time intervals over the course of a dust generator run for each sample.

To determine whether grinding affects the analytical results for a sample, an analysis of variance (ANOVA) was conducted. The purpose of the ANOVA was to test whether the observed differences in analytical results between ground and unground pairs was greater than the variation observed between replicate analyses of filters derived from each of the individual (ground or unground) samples. Measured concentrations derived from each replicate analysis of each individual sample are reported in Table 2-2.

Table 2-2:
Asbestos Concentrations Measured in Replicate Preparation of Selected Samples

	Asbestos Protocol Structure Concentrations (S/g_{PM10})		
Sample Number	**Concentration in Replicate 1**	**Concentration in Replicate 2**	**Concentration in Replicate 3**
Sample Number 1 (unground)	2.30×10^7	2.03×10^7	3.60×10^7
Sample Number 1 (ground)	1.94×10^7	5.90×10^6	----
Sample Number 2 (unground)	1.88×10^7	2.46×10^7	----
Sample Number 2 (ground)	2.06×10^7	6.09×10^6	----
Sample Number 3 (unground)	2.17×10^7	5.88×10^6	----
Sample Number 3 (ground)	1.90×10^7	3.90×10^6	----

	Asbestos PCME Structure Concentrations (S/g_{PM10})		
Sample Number	**Concentration in Replicate 1**	**Concentration in Replicate 2**	**Concentration in Replicate 3**
Sample Number 1 (unground)	6.5×10^7	7.3×10^7	5.4×10^7
Sample Number 1 (ground)	1.04×10^8	1.11×10^8	----
Sample Number 2 (unground)	9.17×10^7	1.48×10^8	----
Sample Number 2 (ground)	1.65×10^8	9.13×10^7	----
Sample Number 3 (unground)	1.19×10^8	1.59×10^8	----
Sample Number 3 (ground)	7.7×10^7	4.2×10^7	----

It is clear from a visual comparison of the measurements presented in Tables 2-1 and 2-2 that variation between ground and unground pairs is no greater than the variation observed between sample replicates. These observations are confirmed formally from the results of the ANOVA in which the variation between ground and unground samples

is shown to be no greater than variation among sample replicates[2]. Thus, measured concentrations for ground and unground pairs do not significantly differ.

Whether measured concentrations differ for ground and unground sample splits was also tested using a more rigorous procedure. In this latter procedure, the individual asbestos structures observed during each analysis were evaluated to determine whether the counts observed among the ground and unground split from each sample could be considered to have been derived from a single Poisson distribution (i.e. could both splits be considered to have come from Poisson distributions exhibiting the same mean, after adjusting for differences in the mass of material deposited on each filter).

This latter evaluation relies on the assumption that asbestos structures are randomly distributed across each sample filter. When this assumption is true, the number of structures observed while scanning a filter is described by a Poisson distribution with a mean equal to the total number of structures on the filter multiplied by the fraction of the surface area of the filter scanned during analysis. Thus, if the only source of variation that contributes to a measurement is the chance of encountering asbestos structures while scanning a fixed area of a filter, the variation observed among repeated measurements over such a filter would be described by a Poisson distribution. This is the type of variation that is observed, for example, among a set of measurements derived by repeatedly scanning randomly selected areas of uniform size on a single filter. Further discussion of Poisson distributions and counting statistics is provided in Section 2.5.

Because the replicate measurements presented in Table 2-2 are derived from multiple filters, additional sources of variation can potentially be introduced by differences in the manner in which asbestos was deposited on each filter. Moreover, ground and unground splits are two physically separate samples that have been separately handled prior to analysis, which suggests further opportunities for introducing variation between such samples. Nevertheless, due to the design of this method, it is expected that the variation between replicate filters (and even sample splits) will be dominated by variation due to counting statistics, which are described by a Poisson distribution (as discussed above).

Results from the more rigorous test to determine whether paired ground and unground splits could be considered to be derived from the same Poisson distribution are somewhat mixed. Among measurements of protocol structures, each pair of ground and unground splits from the three samples can be adequately described by a single Poisson. Among measurements of PCME structures, the ground and unground pair

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[2] The F-test statistics (which are the ratios of the variance between ground and unground samples relative to the variance within ground and unground samples, respectively) from the ANOVA are: $F = 2.55$, $P = 0.15$ (not significant) for protocol structures and $F = 0.14$, $P = 0.72$ (also not significant) for PCME structures.

representing Sample No. 2 can be adequately described by a single Poisson. However, the pairs representing Sample Nos. 1 and 3, respectively, cannot be adequately described by a single Poisson.

The reason for deviation from Poisson counting statistics among the splits for Sample Nos. 1 and 3 (based on counts of PCME structures) is unclear. It may be due to additional contributions to variation (from the kinds of sources described above) for these specific samples. This would make the test for compliance with Poisson counting statistics too severe for the purpose intended here. Overall, however, the results of the statistical tests described above indicate that grinding a sample during preparation has little or no effect on the outcome of a measurement derived using this method.

Given the above, it is recommended for this method that mechanical modification of bulk materials of interest be limited to the minimum that is required to generate 40 g to 70 g samples containing individual particles no larger than approximately 1 cm (3/8ths in.) in diameter. Thus, unconsolidated material need only be sieved (to separate out particles larger than 1 cm), homogenized, and split (Chapter 7). Rocks or other bulk solids may additionally need to be coarse crushed so that the majority of the fragments can pass through a 1 cm sieve.

It is expected that, as long as mechanical crushing, grinding, or other disaggregation is not so severe as to cause degradation of the embedded asbestos structures themselves and as long as any such mechanical actions are performed in a manner allowing capture and preservation of the resulting fines (as part of the sample), then preparation employing such activities should not substantially affect analytical results derived using this method. However, additional studies of such effects over a broader range of sample types and conditions than those reported above would certainly improve our understanding of the capabilities of this method.

2.3 ACCURACY

Because there are no bulk standards available for asbestos, it is not currently possible to determine the accuracy of this method by direct means. However, an indirect procedure exists that can be used to provide a bounding estimate of the accuracy of measurements derived using this method.

Controlled experiments in which bulk materials in the field are subjected to mechanical disturbance and the airborne asbestos concentrations that result from such disturbance are measured, provide an independent set of measured airborne concentrations that can be compared with concentrations predicted by combining bulk measurements using this method with appropriately matched emission and dispersion models. The degree of agreement between measured and predicted airborne concentrations then provides

a measure of the accuracy of such predictions. In turn, the accuracy of such predictions represent bounding estimates for the accuracy of this method.

Such a study has been reported in a recent publication (Berman 2000). Results from this study suggest that airborne asbestos concentrations predicted based on single measurements of bulk concentration (derived from a composite of samples collected from an appropriately designed sampling array) are likely to be accurate to within a factor of three or four. Moreover, the accuracy of predictions for long-term average exposure concentrations (which is what is required to support risk assessment) should be even better. The precision of predictions derived from method measurements can also be improved by basing such predictions on the mean of multiple bulk measurements, rather than single measurements (see Section 2.5).

Importantly, although the results discussed above are suggestive, they are limited. The Berman (2000) study is a single study of two serpentine surfaced roads. The range of conditions examined in this study was limited. Among published dust models that might be coupled with measurements using this method to predict airborne asbestos concentrations, the dust model employed for unpaved roads (U.S. EPA 1985) in the Berman study is among the most highly developed, best tested, and most precise. Moreover, the study was conducted under conditions that were particularly favorable to control of meteorological effects and other potentially confounding variables. Thus, the accuracy of predictions derived for other exposure scenarios may not be as good. Nevertheless, it is apparent from the Berman (2000) study that the accuracy of exposure predictions that are derived in the manner described above will be limited primarily by the accuracy of the dust model used for deriving the predictions, rather than from the input measurements derived using this method.

Additional studies similar to Berman (2000) are recommended in which a broader range of release scenarios are evaluated under a broader range of meteorological conditions. Such studies would allow better characterization of the performance of this method and the predictions derived by modeling that employ measurements using this elutriator method.

2.4 SENSITIVITY

A range of target sensitivities for this method were estimated from calculations presented in the feasibility study (Berman 1990) for the original Superfund Method (Berman and Kolk 1997). However, such targets were based on assumed preparation by an indirect transfer procedure, which is no longer recommended. The original sensitivity calculations were also based on procedures for evaluating asbestos-related risks that are not consistent with the new protocol (Berman and Crump 1999b).

Therefore, a new set of target sensitivities has been estimated based on recent calculations (Berman unpublished). In these calculations, acceptable airborne concentrations for protocol structures were separately estimated for chrysotile and the amphiboles based on a target risk level of 10^{-5} (one in one hundred thousand) and the procedures recommended for relating exposure and risk that are presented in the new protocol (Berman and Crump 1999b).[3]

Based on the unpublished calculations, a target sensitivity of 3×10^6 S/g_{PM10} is likely to adequately bound the range of concentrations of potential concern for the vast majority of emission and dispersion scenarios of interest for risk management. However, to assure adequate sensitivity for specific projects, it is highly recommended that preliminary calculations be performed to identify a level of sensitivity that will adequately support risk management decisions that are project specific. Therefore, the target sensitivity presented here is intended to be illustrative only.

> Note that the target sensitivity provided in the last paragraph is based on calculations assuming amphibole asbestos protocol structures. Because chrysotile appears to be substantially less potent than the amphiboles, a target sensitivity on the order of 5×10^7 S/g_{PM10} may generally prove sufficient for chrysotile asbestos. Again, however, sensitivity for this method should be set based on project-specific requirements and the stopping rules for analysis (Section 10.1.2) should then be adjusted accordingly.

As indicated in Section 10.1.2, achieving the lower (more severe) of the two sensitivities listed above should ultimately require scanning a maximum of approximately 150 grid openings (distributed over 5 grid specimens), when filter preparation during dust generation is optimized. Because scanning need only be performed at a maximum magnification of 10,000x, the cost for performing such an analysis is expected to remain reasonable (see Section 2.8).

2.5 PRECISION

The original Superfund Method was shown to achieve a level of precision that is adequate for distinguishing acceptable asbestos concentrations from potentially hazardous concentrations with a resolution that is considered acceptable for supporting risk management decisions (Berman et al. 1994). During the pilot study, the counting of 50 structures (during the analysis of paired splits from each of several homogenized,

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[3] There are six different mineral types included in the definition of asbestos that are grouped into two mineralogical categories: serpentine asbestos (chrysotile) and the amphiboles, which include the other five types of asbestos. Asbestos structures in each of the two mineralogical categories have been found to exhibit differing potency (Berman and Crump 1999a). That is why the new risk assessment protocol incorporates differing risk factors for chrysotile and the amphiboles (Berman and Crump 1999b).

bulk samples) produced an average relative percent difference (RPD) across duplicate sample pairs of 47%. Of 40 paired counts (five separate counts of each of a pair of filters from each of four sets of paired samples by each of two laboratories), 14 (35%) exceeded an RPD of 50% and 6 (15%) exceeded an RPD of 100%. However, 8 of the 14 RPD's exceeding 50% derive from a single set of paired splits so it is conceivable that special problems developed during the preparation of this one pair of splits. For further discussion of this issue, see Berman (2000).

Importantly, the results presented above were derived based on analysis of samples prepared using an indirect transfer technique. Because this method relies primarily on the analysis of samples prepared by direct transfer, additional tests were recently performed to determine the achievable precision for the analysis of samples prepared for TEM analysis by direct transfer (Berman unpublished).

In a recent series of studies, replicate filters were collected during dust generator runs from each of a series of 10 samples. The filters were then prepared for TEM analysis using a direct transfer technique and each of two types of asbestos structures (protocol structures and PCME structures) were counted. Measurements were then evaluated to determine whether counts derived from replicate filter pairs could be adequately described by a single Poisson distribution (after adjusting for differences in the mass deposited on each replicate).

The results of this evaluation indicate that, for protocol structures, counts derived from replicate filters cannot be distinguished from one another (i.e. they can be adequately described by a single Poisson distribution) for 8 of the 10 pairs tested. Although the replicates from the remaining two samples could be distinguished at the 95% level of significance, they were indistinguishable at the 99% level of significance (P = 0.039 and 0.045 for the two pairs, respectively). Thus, the dominant source of variation apparent among these replicates is that attributable to counting (see Section 2.2), which is the minimum variation achievable for asbestos analyses that require the enumeration of individual asbestos structures.

Results for the 9 available pairs of replicates analyzed for PCME structures showed somewhat greater variability, but generally support the same conclusion. Of the PCME structure counts derived from replicate filters 6 of 9 replicate pairs cannot be distinguished from one another (i.e. each pair of replicates can be adequately described by a single Poisson distribution). Two of the remaining three pairs of replicates can be distinguished at the 95% level of significance but not at the 99% level of significance (P = 0.042 and 0.016, for the two pairs, respectively). The two filters of the last replicate pair exhibit structure concentrations that are clearly distinguishable from one another (they cannot reasonably be described by a single Poisson, P = 0.008). It is not known why counts of PCME structures appear to be somewhat more variable than counts of protocol structures. Overall, however, it is reasonable to

conclude from these data that the variability observed between replicate measurements derived from this method is dominated by that attributable to counting. This means that the precision of the method approaches the optimum achievable for a method that involves the enumeration of individual asbestos structures.

> Note: the above described test of precision (for samples prepared by direct transfer) is based on the analysis of replicate filters rather than paired splits of homogenized samples (which would address additional sources of variation potentially introduced by the handling of bulk samples prior to dust generation). It is expected, however, that the achievable precision for paired sample splits will be comparable to that demonstrated for replicate filters (i.e. variation will be dominated by the limitations of counting statistics and will therefore approach the minimum possible). This is indicated by the results of the original pilot study (Berman et al. 1994), which demonstrate that variation introduced by bulk sample handling in this method is trivial compared to the variation associated with filter generation and the variation introduced by counting statistics during analysis. Both of the latter sources of variation are adequately addressed by the comparison of results from replicate filters.

2.6 OBJECTIVE PROCEDURES FOR SAMPLE HANDLING AND ANALYSIS

This method is designed to be adopted and applied broadly. Therefore, with one exception, equipment and supplies required to implement this method are commercially available and in common use among laboratories offering analyses for the determination of asbestos by TEM.

The one exception involves the need to construct a dust generator to conduct the required sample preparation. However, even the dust generator is designed to be constructed from commercially available materials and precise specifications for its construction have been published in the Superfund Method (Berman and Kolk 1997). The specifications for the modifications required to adapt the dust generator for use with this method are incorporated within this document (Section 6.3). The cost for constructing the required dust generator has proven to be modest and should not be a hindrance to any laboratory wishing to offer commercial analyses using this method.

So that laboratories offering analysis using this method will generate comparable results, the procedures incorporated into the method were designed and selected to facilitate objective implementation and are robust (i.e. relatively insensitive) to subjective application that might otherwise introduce variability within and between laboratories. The data and evaluation presented in Section 2.2 provides a good indication of the degree to which the procedures incorporated into this method for

sample preparation are robust to subjective application. Procedures employed for analysis are also intended to limit the effects of subjective application.

While it is unavoidable that the counting rules employed to determine the manner in which asbestos structures are to be enumerated during analysis are subject to analyst interpretation and this introduces some variation, it is expected that such variation will be somewhat more limited for this method due to the constraints in the range of structure sizes that are included for analysis. Structures counted during analysis by this method are limited to those defined as protocol structures (Section 2.1). At the same time, subjective variation introduced by the interpretation of counting rules is a limitation that is common to all TEM methods used for the determination of asbestos structure concentrations.

The need to produce filters during dust generation with dust deposits that are demonstrably uniform must also be considered when addressing the reproducibility of repeated analyses and the comparability of analyses within and between laboratories.

To test this ability initially, five filters were selected for evaluation from among filters generated during a series of recent studies. Multiple grid specimens (up to nine for some filters) were then prepared from locations on the filter selected in systematic arrays designed to representatively cover the entire surface of each filter. Asbestos structures were then enumerated during scans of multiple grid openings (up to 50) on each of the grid specimens that were prepared from each filter. The resulting structure counts were then evaluated to determine whether the counts across grid openings within each grid specimen and, separately, whether the total counts across grid specimens from each filter could be adequately described by a single Poisson distribution.

Results from this evaluation indicate that counts on grid openings within every grid specimen tested could always be adequately described by a single Poisson distribution. However, only three of the five filters yielded a set of grid specimens that could be adequately described by a single Poisson. This was not a problem as long as samples were being prepared by an indirect transfer procedure. However, direct transfer is now preferred, due to research indicating superior correlation with risk (Berman and Crump 1999a).

After further study, it was found that lack of uniformity on the filters prepared for asbestos analysis using the dust generator was primarily due to the lack of complete mixing between the air stream exiting the tumbler of the dust generator and the air stream entering from the open tube at the bottom of the elutriator (in the original configuration of the dust generator). In hindsight, the laminar flow regime of the elutriator is not conducive to efficient mixing of such air streams.

Based on these results, the dust generator was reconfigured by eliminating the open tube at the bottom of the elutriator and redesigning to assure adequate flow velocities at the bottom of the elutriator[4] to assure that respirable particles are transferred quantitatively from the tumbler to the elutriator (see Section 6.3). Based on visual inspection of filters that are intentionally overloaded so that the deposit on the filter is readily visible, it appears that the reconfigured dust generator produces adequately uniform deposits on filters so that there should be no problem demonstrating that structures observed on such filters are Poisson distributed. Further analysis of asbestos structure counts are in progress.

2.7 THE CHARACTERISTICS OF BULK MATERIALS REQUIRING ANALYSIS

The asbestos content of a broad range of bulk materials (both natural and anthropogenic) are expected to provoke interest. These may include, for example:

- rock or soils containing naturally occurring asbestos;
- soils, fills, or sediments containing asbestos-containing debris;
- asbestos-containing construction materials; and
- asbestos-containing, settled dust.

This method is designed to be applied broadly and the procedures provided herein should allow the method to be adapted for application to any of the kinds of materials listed above. For descriptions of adaptations that may be required for some of the materials listed, see Sections 2.2, 8.2, and 8.3.

To date, the original Superfund Method and this modified elutriator method have been applied to a broad range of asbestos containing materials of interest including:

- crushed serpentine rock variously containing naturally occurring chrysotile and tremolite asbestos;
- soils variously containing naturally occurring chrysotile and tremolite asbestos; and

[4] Air stream velocities at the bottom of the elutriator must be higher than velocities in the main body of the elutriator at all points in the air stream so that respirable particles are not lost prior to sorting in the elutriator. This assures quantitative transfer of respirable material from the tumbler to the filters where dust and asbestos are collected for analysis.

- soils and fills variously containing chrysotile, amosite, anthophyllite asbestos, tremolite asbestos, and actinolite asbestos in construction debris.

The concentrations of asbestos that have been observed in samples analyzed using this method to date (excluding non-detects) range between 1 x 10^6 S/g_{PM10} and 8 x 10^8 S/g_{PM10} for protocol structures and as high as 4 x 10^9 S/g_{PM10} for total structures.

2.8 COST CONSIDERATIONS

Per-sample costs for analysis using the original Superfund Method tend to run between $800 and $1,500, depending on the target sensitivity and precision requested for a particular analyses. Given the modifications adapted for this method, a target sensitivity of 3 x 10^6 S/g_{PM10}, and counts that are restricted to protocol structures, per-sample costs for analysis using this method have been running between $500 and $800 (approximately half of per-sample costs for the original method). As experience with this method increases and as competition increases with additional laboratories offering this method commercially, it is also expected that per-sample costs for this method will decrease further.

Importantly, per-sample costs for analysis using this method are not expected to represent a driving factor for the cost of site characterization employing this method. The procedures incorporated into this method for sample collection and field preparation are amenable to substantial compositing so that, when an investigation is properly designed, only a very limited number of analyses using this method should be required to provide adequate characterization of the bulk materials of interest. In fact, the process of combining results from this method with appropriately selected dust emission and dispersion models to predict airborne exposure concentrations is expected to be less expensive than characterizing airborne exposures by direct measurement (with a comparable degree of accuracy) for all but the simplest cases of potential interest.

3.0 OVERVIEW OF METHOD

Samples are collected in the field according to a pre-defined sampling plan identifying the number of samples to be collected and the locations from which samples are to be collected. Procedures for designing such a plan are beyond the scope of this document but are reported elsewhere (see, for example, Berman and Chesson, unpublished).

Any of a variety of commercially available sampling equipment (i.e. trowels, shovels, augers, corers, etc.) may be used to collect samples for this method. However, they must have been specified in the pre-defined sampling plan based on the nature of the material being sampled and the depths over which samples are to be collected. Whatever sampling technique is employed, the *minimum* size sample to be collected at each location shall be 1 kg.

Once collected, each sample is brought to a central location for field preparation. Field preparation steps are listed in Figure 3-1 and discussed further in Chapter 7 and in greater detail in Chapter 8 of Berman and Kolk (1997). Each sample is first weighed. Then the sample is sieved using a screen with 3/8th in. (1 cm) openings to separate a coarse and fine fraction. The material placed on the sieve is worked with gloved hands to assure that all friable components pass through the screen.

The coarse fraction, composed of material that is retained by the screen, is transferred to a bucket and weighed prior to discarding on site. The fine fraction is also weighed. As indicated in Figure 3-1, the fine fraction is then homogenized. The procedure recommended in this method for homogenization is repetitive splitting using a riffle splitter with the split halves of the sample being re-combined at the end of each split. Studies indicate that five to seven iterations are typically sufficient to achieve adequate homogenization.

Once homogenized, the fine fraction is then sub-sampled using the riffle splitter (Figure 3-1). During sub-sampling, the one-half of the sample from one of the two receiving trays is discarded after each split and the second half of the sample is then re-split. The process is repeated until sub-samples weighing between 40 g and 70 g are produced in each of the two receiving trays. The material in each tray is then transferred quantitatively to a sample bottle, packaged and shipped to the laboratory.

Sample handling, preparation, and analysis in the laboratory is depicted in Figure 3-2 and described in detail in Chapter 8 (of this document). Once sub-samples weighing between 40 and 70 g are obtained, they can be separately prepared and analyzed (Section 8.5).

To prepare samples, as indicated in Figure 3-2, first load the sample into the tumbler of a dust generator. The design, construction, and operation of a dust generator suitable for use with this method is provided in Appendix A of Berman and Kolk (1997) with required modifications provided in Section 6.3 of this document. The sample is then conditioned by flowing humidity-controlled air through the tumbler and over the sample for several hours.

Once the sample is conditioned, the tumbler of the dust generator is started and a sample run is initiated. During each run, a series of filters is collected continuously from the top of ME openings of the dust generator and these are weighed and plotted to determine when dust generation has stabilized sufficiently to collect filters for asbestos analysis.

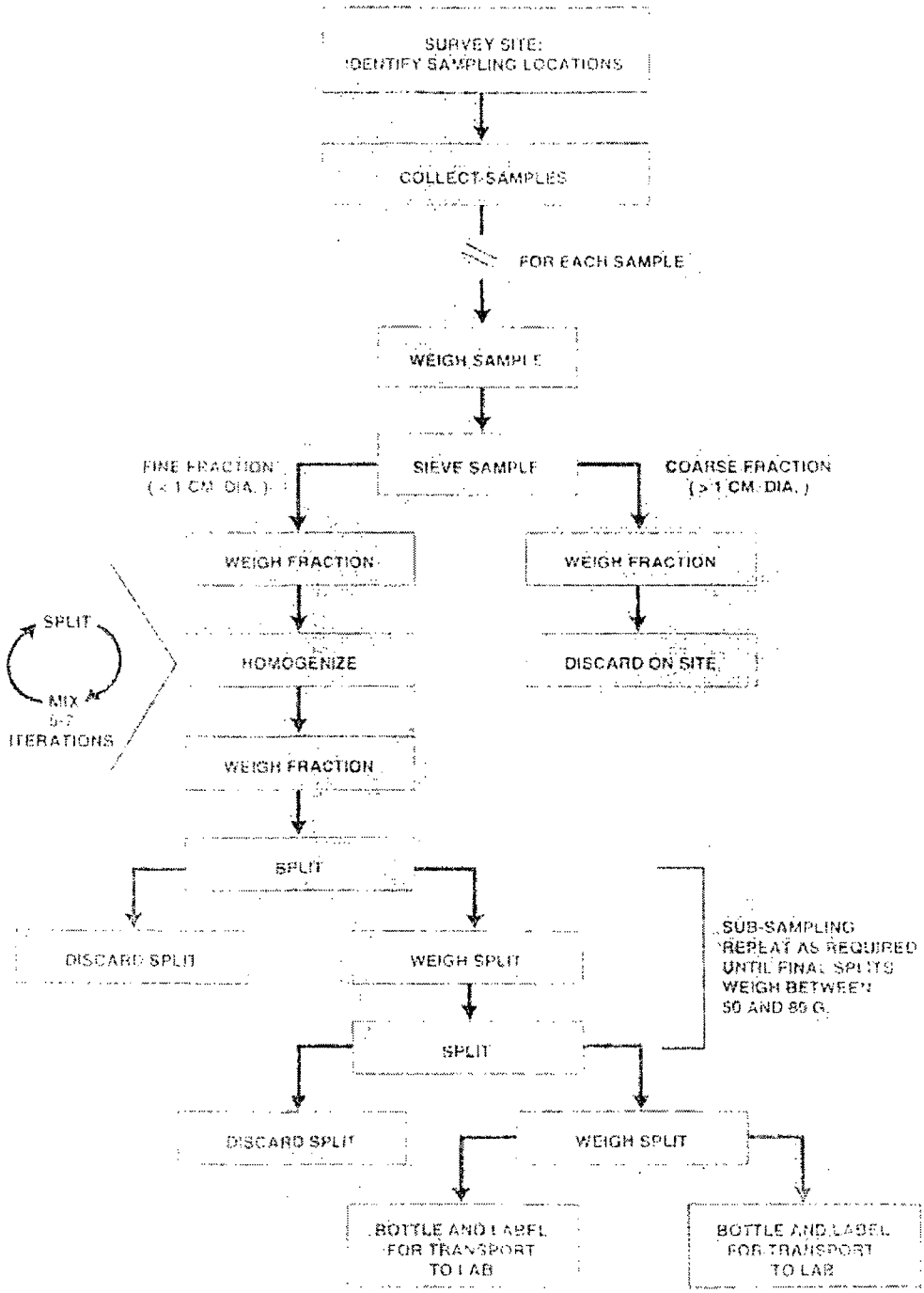
While the dust generator is operating, a second set of filters is also collected over the IST opening of the dust generator, which articulates with an isokinetic sampling tube. These are collected such that loading is appropriate for specimen grid preparation using a direct transfer technique.

Next, as indicated in Figure 3-2, TEM specimen grids are prepared using a direct transfer technique from the filters collected from atop the isokinetic sampling tube of the elutriator. Before specimen grid preparation, however, the filters must be weighed to determine the mass of dust deposited on the filter. Specimen grids are then analyzed using the counting and identification rules of the International Standards Organization (ISO) Method for the determination of asbestos in air using an indirect transfer technique (ISO 10312) with the counting rules modified for determining only protocol structures (defined in Section 10.1.1) and with the stopping rules modified as indicated in Section 10.1.2.

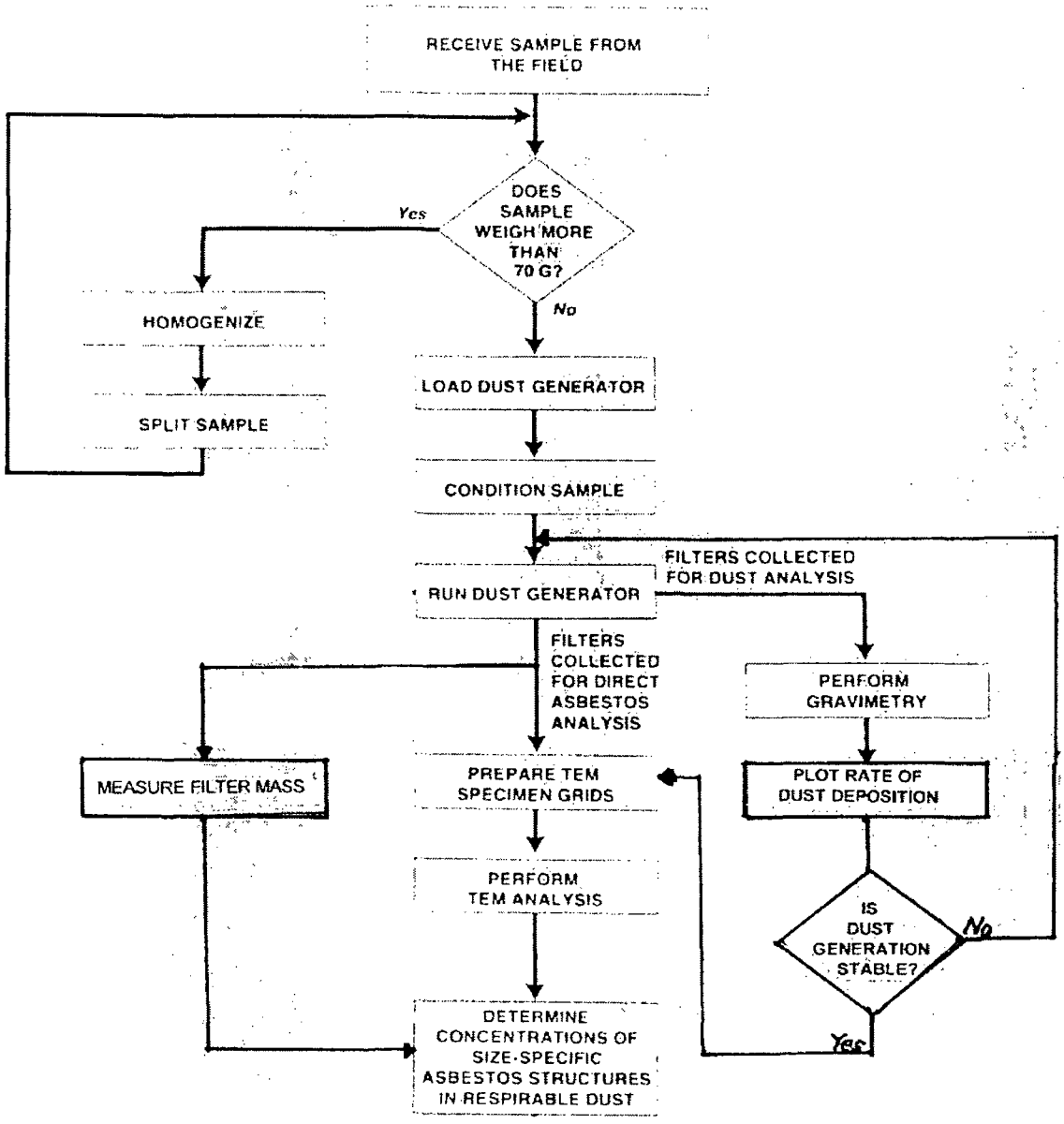
Dust mass estimates are then combined with asbestos counts to allow reporting of the concentration of asbestos protocol structures per unit mass of respirable dust in the sample (Figure 3-2).

FIGURE 3-1

SAMPLE COLLECTION AND FIELD PREPARATION



LABORATORY PREPARATION AND ANALYSIS



4.0 DEFINITIONS

Amphibole: a group of rock-forming ferromagnesium silicate minerals, closely related in crystal form and composition, and having the nominal formula:

$$A_{0-1}B_2C_5T_8O_{22}(OH,F,Cl)_2$$

where:

A = K, Na;

B = Fe^{2+}, Mn, Mg, Ca, Na;

C = Al, Cr, Ti, Fe^{3+}, Mg, Fe^{2+};

T = Si, Al, Cr, Fe^{3+}, Ti.

In some varieties of amphibole, these elements can be partially substituted by Li, Pb, or Zn. Amphibole is characterized by a cross-linked double chain of Si-O tetrahedra with a silicon:oxygen ratio of 4:11, by columnar or fibrous prismatic crystals and by good prismatic cleavage in two directions parallel to the crystal faces and intersecting at angles of about 56° and 124° (Hodgson 1965).

Amphibole Asbestos: amphibole in an asbestiform habit.

> Note, based on work performed to define the relationship between structure morphology and health effects (Berman and Crump 1999a), it appears to be primarily the overall dimensions of an asbestos structure that determine it's potential to contribute to disease, not whether that particular structure is strictly "asbestiform" in habit (see the definition of asbestiform below). Therefore, prudence dictates that all isolated amphibole asbestos structures that exhibit dimensions consistent with those defined as "protocol structures" should be included in the determination of risk-related concentrations (see the definition of protocol structures).

Analytical Sensitivity: the calculated asbestos concentration in soil or a bulk matrix, in asbestos structures per gram of respirable dust (S/g_{PM10}), equivalent to counting of one asbestos structure in the analysis.

Asbestiform: a specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.

> Note, the term, "asbestiform" is primarily a geology term that is intended to distinguish deposits of a particular mineral that exhibit high fibrosity *as a bulk property* in contrast to massive deposits in which the material is variously described as columnar, platy, or granular (Kraus et al. 1959). The term was not originally intended to distinguish the potential for a material to contribute to health effects. Therefore, this distinction should not be applied to isolated asbestos structures when determining risk-related concentrations (see the note under the definition for amphibole asbestos). While it appears there is generally good correlation between an asbestos mineral's habit and the potential to induce adverse health effects (see, for example, OSHA 1992), the correlation may not be perfect (see the definition of protocol structures).

Asbestos: a term applied to a group of fibrous silicate minerals that readily separate into thin, strong fibers that are flexible, heat resistant and chemically inert. The term is generally applied to asbestiform serpentine and the non-aluminous amphibole minerals (Kraus et al. 1959).

> Note, the term, "asbestos" is a geology term that was originally intended to distinguish the bulk properties of deposits with potential commercial value (due to fibrosity) from those that do not exhibit such properties. It was not initially intended to define materials that present a risk for inducing adverse health effects (when inhaled). When assessing health effects, the term asbestos should be considered to include all isolated structures of the proper mineralogy that exhibit a morphology consistent with that defined below as "protocol structures."

Asbestos Component: a term applied to any individually identifiable asbestos sub-structure that is part of a larger asbestos structure.

Asbestos Structure: a term applied to any contiguous grouping of asbestos fibers, with or without equant particles.

Aspect Ratio: the ratio of the length to width of a particle.

Blank: a fiber count made on TEM specimen grids prepared from an unused filter (or a filter through which asbestos-free air or water has been passed), to determine a background measurement. Blanks may include equipment blanks, field blanks, filter or lot blanks, and laboratory blanks, and/or method blanks.

Bundle: a fiber composed of parallel, smaller diameter fibers attached along their lengths (ISO 10312).

Chrysotile: the asbestiform habit of a mineral of the serpentine group that has the nominal composition:

$$Mg_3Si_2O_5(OH)_4$$

In some varieties of chrysotile, the silicon may be partially substituted by Al or less commonly by Fe. The magnesium may be partially substituted by Fe, Ni, Mn or Co. Some varieties contain Na, Cl or both. Chrysotile is a highly fibrous and silky variety and constitutes the most prevalent type of asbestos (Hodgson 1965). Also, see notes under the definitions for asbestiform, asbestos, and protocol structures when considering the health effects potentially attributable to chrysotile.

Cluster: an assembly of randomly oriented fibers (see Chatfield 1993).

Component Count: for any sample, a tally that includes the individually identified components of complex asbestos structures and each single asbestos structure with no identifiable components.

Conditioning Filters: a collected at the beginning of a run while a sample is being conditioned. Under such circumstances, the tumbler is loaded with sample but is not running. However, humidity controlled air is being passed through the tumbler, through the elutriator, and into the filter mounts.

Elutriator: a device in which differential flow through a fluid (gas or liquid) against an opposing force (i.e. gravity) is employed to separate particles by size.

Equant Particle: as used in this document, a non-asbestos particle bound to, or overlapping with, asbestos structures observed on a TEM specimen grid.

Equipment Blank: a filter collected from a mount over one of the openings atop the elutriator while air is passed through an empty tumbler assembly and the elutriator.

Fiber: an elongated particle that has parallel or stepped sides. In this method, fibers that potentially contribute to biological activity are restricted to those exhibiting dimensions consistent with protocol structures (see Berman and Crump 1999b).

Fibril: a single fiber of asbestos that cannot be further separated longitudinally into smaller components without losing its fibrous properties or appearance.

Fibrous Structure: a contiguous grouping of fibers, with or without equant particles.

Field Blank: a bulk material known to be free of asbestos that is packaged in such a manner so as to be indistinguishable from other field samples (for a particular project) and is sent to the laboratory for preparation and analysis using this method.

Filter Blank: an unused filter that is analyzed to determine the background asbestos structure count on the filter matrix.

Friable: as used in this document, capable of being crushed or deformed with the hand with the attendant release of fibers.

Habit: the characteristic crystal form or combination of forms of a mineral, including characteristic irregularities.

Identify: during asbestos analysis, the use of a sequential set of procedures to determine and confirm the mineralogy of a structure.

Isokinetic Sampling: sampling air in such a manner so as not to disturb the direction or velocity of air flow at the point sampled.

Isokinetic Sampling Tube: a tube placed in the air flow of the vertical elutriator portion of the dust generator used in this method, which samples the air at the top of the elutriator isokinetically.

Laboratory Blank: an unused filter that is analyzed along with sample filters to determine the background asbestos structure count in laboratory air.

Lot Blank: see filter blank.

Matrix:[5] A connected assembly of asbestos fibers with particles of another species (non-asbestos) (ISO 10312).

Method Blank: a filter collected in a mount over one of the openings atop the elutriator while washed, play sand is tumbling under conditions appropriate for a routine sample run.

PCM Equivalent Structure: a structure of aspect ratio greater than or equal to 3:1, longer than 5 µm, and which has a mean diameter between 0.2 µm and 3.0 µm for a part of its length greater than 5 µm. PCME structures also must contain at least one asbestos component (see Berman and Chatfield 1999a).

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[5] The above definition applies when used to describe an asbestos structure. The term is also used in this document to describe a heterogeneous bulk solid.

Note that the definition of PCME structures is provided here primarily for historical perspective.

Protocol Structure: an isolated structure or a component of a complex structure that is longer than 5 µm and thinner than 0.5 µm. When determining risk-related concentrations, protocol structures longer than 10 µm must be distinguished and separately enumerated because such structures are assigned a greater potency than protocol structures with lengths between 5 µm and 10 µm (see Section 2.1). When counting protocol structures to assess health effects, both isolated parent structures exhibiting the appropriate dimensions and qualifying components of more complex structures should be included in the count.

Note, it has been observed that only a small fraction of the isolated particles produced by the degradation of minerals found in a massive habit qualify as protocol structures. In contrast, minerals found in an asbestiform habit tend to yield large numbers of protocol structures when mechanically or naturally disturbed. Therefore, the relative hazard associated with the asbestiform versus massive habits of a mineral may be related largely to the relative rate of release of (and the attendant levels of exposure to) protocol structures when such materials are disturbed under comparable conditions. Moreover, under conditions typically associated with quarrying and mining of materials containing small quantities of the massive forms of the asbestos-related minerals, the exposures attendant to the disturbance of such materials are generally not considered sufficient to warrant concern other than as nuisance dusts (OSHA 1992). Incidentally, protocol structures are defined in a manner that is consistent with the requirements recommended by Wylie to OSHA for distinguishing among structures that derive from asbestiform and non-asbestiform habits, respectively (OSHA 1992, P. 24329).

Respirable Dust: particulate matter in a size range capable of penetrating the deep lung and being deposited in terminal bronchioles and alveoli (beyond the reach of the ciliary escalator, see Berman and Crump 19991). This is further defined as particulate matter exhibiting an aerodynamic equivalent diameter less than 10 µm (Raabe 1984). The aerodynamic equivalent diameter of a particle is the diameter of a hypothetical spherical particle of unit density that exhibits the same settling velocity in air as the particle of interest. Due its size range, respirable dust is commonly abbreviated as PM_{10}.

Riffle Splitter: a device composed of a hopper and multiple, uniform, parallel chutes that alternately feed from the hopper to opposing receiving trays.

Scrubber: a device for removing particles from an air stream by passing the air stream through a super-saturated vapor in which the particles serve as nudeation centers for

condensation and are thus captured. The resulting droplets (containing the trapped particles) then fall back into a central reservoir of boiling liquid.

> Note that among the modifications to the dust generator from the Superfund Method that is defined in this modified elutriator method is to remove the scrubber from the dust generator assembly and to seal the ports for the scrubber with stoppers (see Section 6.3). Therefore, this definition is provided primarily for historical reference.

Serpentine: a group of common rock-forming minerals having the nominal formula:

$$Mg_3Si_2O_5(OH)_4$$

Serpentine deposits often contain chrysotile asbestos (which is serpentine in an asbestiform habit).

Silt: the fine material found in a bulk matrix. Silt is defined formally as material less than 70 µm in diameter.

Structure Count: for any sample, a tally of each individually identified asbestos structure regardless of whether the structure contains identifiable components. This is equivalent to a count of the total number of separate asbestos entities encountered on the sample.

Vertical Elutriator: see Elutriator.

Tumbler: a device that is rotated to provide continuous agitation to a bulk material placed inside. In the dust generator employed in this method, air is blown through a tumbler containing sample to carry away the dust generated during agitation by the tumbler.

> Note that the tumbler incorporated into the dust generator that is required for sample preparation in this method is designed primarily to facilitate capture of respirable size particles in the air stream that flows through the tumbler. It is not intended to alter the bulk characteristics of the sample.

Washed, Play Sand: a commercially available material composed of well-sorted sand that contains virtually nothing fine enough to be considered respirable.

5.0 SYMBOLS AND ABBREVIATIONS

5.1 SYMBOLS

A_f - the area of a filter from which a specimen grid is prepared (mm^2).

A_{go} - the average area of a specimen grid opening (mm^2).

C_{dust} - the concentration of asbestos structures (of a defined size and type) in the respirable dust from a sample (S/g_{pm10})

C_{mtrx} - the concentration of asbestos structures (of a defined size and type) in the original field matrix that was sampled for analysis using this method.

C_{smpl} - the concentration of asbestos structures (of a defined size and type) in a soil or bulk sample (S/g).

cm - centimeter (10^{-2} meter).

cm^2 - square centimeter.

cm^3 - cubic centimeter.

cm^3/min- cubic centimeter per minute.

d - the density of a particle (g/cm^3).

D_{rate} - the estimated rate of deposition on a specific filter (g/s).

°C - degrees centigrade.

°K - degrees Kelvin.

ΔM_f - the mass of respirable dust collected on a single filter during the interval Δt (g).

ΔM_s - the mass of respirable dust released from the sample during the interval Δt (g).

Δt - a short time interval (no more than 20 minutes).

η - the dynamic viscosity of air (g/cm*s).

eV - electron volt.

F_c - the percent of airflow (i.e. the % of the volumetric flow rate) through the top exit (ME) opening of the elutriator that does not pass through the isokinetic sampling tube (%).

F_d - the percent of airflow (i.e. the % of the volumetric flow rate) through the top exit (IST) opening of the elutriator that passes through the isokinetic sampling tube (%).

ft - foot.

g - gram.

g - the acceleration due to gravity (cm/s^2), when used as a variable in an equation.

g/L - gram per liter.

g/cm^3 - gram per cubic centimeter.

hp - horsepower.

k - the first order rate constant (s^{-1}).

kg - kilogram (10^3 gram).

kV - kilovolt.

in - inch.

L - liter.

L/min - liters per minute.

M_{coarse} - the mass of the coarse fraction of a matrix sampled in the field.

M_{DEP} - the target deposition loading for an analytical filter.

M_f - the cumulative mass of respirable dust collected on filters from the start of a run to time, t (g).

M_{fine} - the mass of the fine fraction of a matrix sampled in the field.

M_o - the mass of respirable dust in a sample at the start of a run (g).

M_r - the cumulative mass of respirable dust released from a sample from the start of a run to time, t (g).

M_s - the mass of respirable dust remaining in a sample during a run but after time, t (g).

M_{sample} - the mass of a sample introduced into the dust generator (g).

M_{tot} - the total mass of respirable dust estimated to reside in a sample (g).

ml - milliliter (10^{-3} L).

mm - millimeter (10^{-3} meters).

mm^2 - square millimeter.

µg - microgram (10^{-6} grams).

µm - micrometer (10^{-6} meters).

N_{go} - the number of grid openings counted during a scan (#).

nm - nanometer (10^{-9} meter).

P_f - the pressure measured at a flowmeter (torr).

P_t - the pressure estimated at an elutriator opening (torr).

%RD - the mass percent of respirable dust in a sample (%).

r - the radius of a particle (cm).

r^2 - the coefficient of determination (also defined as the correlation coefficient squared).

$R_{a/d}$ - the ratio of the number of asbestos structures (of a defined size and type) to the mass of respirable dust (S/g_{PM10}).

R_f - the flow reading from a flowmeter (cm/s).

R_{slt} - the mass fraction of silt in a bulk matrix (g_{silt}/g_{matrix})

S - the number of asbestos structures.

S_c - the number of asbestos structures (of a defined size and type) counted during a scan (#).

S_d - the number of asbestos structures that must be detected during a TEM scan for asbestos to be defined as detected (#).

S_{anal} - the required analytical sensitivity for this method (defined separately for total and long protocol structures) (S/g_{PM10}).

s - second.

S/g - structures per gram.

S/g_{PM10} - structures per gram of respirable dust.

S/L - structures per liter.

S/mm^2 - structures per square millimeter.

t - time (s).

t_{opt} - the time required to deposit a mass of M_{dep} on a filter (s).

T_f - the temperature at a flowmeter (°K).

T_t - the temperature at an exit opening of the elutriator (°K).

V_l - linear air flow rate (cm/s).

V_v - the volumetric air flow rate (cm^3/s).

W - watt.

5.2 ABBREVIATIONS

ANOVA- Analysis of Variance

ED - Electron diffraction

EDXA - Energy dispersive X-ray analysis

FWHM - Full width at half maximum

HEPA - High efficiency particle absolute

IST - refers to the opening at the top of the elutriator that is associated with the *isokinetic sampling tube*

MCE - Mixed cellulose ester

ME - refers to the *main exit* opening at the top of the elutriator, which is *not* associated with the isokinetic sampling tube

PC - Polycarbonate

PCM - Phase contrast optical microscopy

PCME- Phase contrast microscopy equivalent

PLM - Polarized light microscopy

PM_{10} - Respirable dust

RPM - Revolutions per minute

SAED - Selected area electron diffraction

TEM - Transmission electron microscopy

TSP - Total suspended particulate

UICC - Union Internationale Contre le Cancer

6.0 FACILITIES AND EQUIPMENT

6.1 SAMPLE COLLECTION EQUIPMENT AND CONSUMABLE SUPPLIES

To complete field sampling per this method, the following field equipment is mandatory:

- survey equipment appropriate to the manner in which sample locations are to be defined per the sampling plan;
- appropriate trowels, shovels, augers, or corers for sample collection per the sampling plan;
- (when sampling surface materials) a 12 in square aluminum template with an 8 in square hole in the center;
- a minimum of three 3-gal plastic buckets;
- a brass or steel sieve with 3/8 in. (1 cm) openings;
- a field balance (with a capacity of 40 kg and capable of achieving a precision of ± 10 g);
- a field balance (with a capacity of 2 kg and capable of achieving a precision of ± 0.2 g)[6]
- a riffle splitter with a minimum of 24, 3/4 in. (minimum size) chutes and three sample trays;
- one L plastic sample containers;
- sufficient plastic coolers to store and ship samples at ice temperature;
- equipment for cleaning sampling tools, including:
  - large buckets and tubs;
  - a container of asbestos-free water;
  - garden sprayers;
  - bio-degradable detergent;
  - assorted asbestos-free rags, sponges, etc.;
  - an air compressor with HEPA filter (optional, for drying equipment);
- field logbook and appropriate custody forms and sample labels;

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[6] If appropriate equipment is available, it is advantageous to use a single field balance to achieve both sets of capacity and precision requirements for field weighing.

- assorted garbage bags, paper towels, and tape;
- Tyvek suits and protective gloves; and
- appropriate equipment for respiratory protection.

6.2 LABORATORY FACILITIES

Laboratories wishing to adopt this method must develop and maintain the following facilities:

- a properly ventilated room for bulk sample handling that is isolated from other room(s) in which air samples are handled and analyzed by TEM. All such facilities must be sufficiently well ventilated to allow preparation of blanks that yield background determinations satisfying the requirements of Section 10.6 of the Superfund air method (Chatfield and Berman 1990); and
- a glove box or equivalent isolation chamber of sufficient size to house a riffle splitter (or other equipment) required for the homogenization and sub-sampling of samples for this method. The glove box or isolation chamber must provide ample room for handling kg size soil or bulk samples while maintaining background concentrations in the outside room air at levels considered acceptable as defined in Section 10.6 of Chatfield and Berman (1990).

6.3 LABORATORY EQUIPMENT

Implementing this elutriator method requires use of the following equipment::

- a dust generator-elutriator constructed per the specifications provided in Appendix A of the Superfund Method (Berman and Kolk 1997) and modified as follows:

– reduction of the size of the exit tube leading from the tumbler to the elutriator (through the tumbler bearing). Insert a 1.75 in. length of standard 0.25 in. O.D. copper tube as indicated in Figure 6-1. The tubing is sealed in place with a 1.5 in. sleeve constructed of 0.25 in. I.D. Tygon tubing, as depicted in the figure. Place the Tygon sleeve flush against inner face of the aluminum end plug. The copper tube extends approximately 0.25 in. into the tumbler body (beyond the end of the sleeve);

– placement of a conical shaped plug that fits into the hole at the bottom of the elutriator, as depicted in Figure 6-2. The plug should fit snugly but remain sufficiently loose to be easily removed;

- extension of the tube leading from the tumbler through the elutriator wall and bending downward toward the bottom of the elutriator. Using a removable attachment, extend the length of the 1.5 in. diameter X 0.625 in. wall stainless steel tubing so that it terminates no more than 0.5 in. from the bottom of the elutriator, as depicted in Figure 6-2. The extension may extend so that it just overlaps the top of the plug, but should leave ample clearance for free flow of air around the lip of the tube and up the outside of the tube into the main body of the elutriator; and

- plugging of the exit tube at the bottom of the elutriator and the side ports that originally led to the scrubber, as depicted in Figure 6-3. Place a No. 4 rubber stopper in each of the two exit ports of the elutriator, as depicted in the figure. Use a No. 8 rubber stopper to plug the hole at the bottom of the elutriator, as depicted in the figure.

  Note that plugging the hole at the bottom of the elutriator obviates the need for the glass cup that was mounted over the bottom hole in the original configuration of the device (Figure A-6 of Berman and Kolk 1997). This also eliminates the small opening in the cup that served as a second source of air into the elutriator in the original configuration (Figure A-1 of Berman and Kolk 1997).

  Note further that all modifications are designed such that the tumbler and elutriator can be returned to their original configuration (described in Berman and Kolk 1997) within a matter of minutes;

- a TEM operating at an accelerating potential of 80-120 kV, with a resolution better than 1.0 nm and a magnification range of approximately 300 to 100,000. The ability to obtain a direct screen magnification of about 100,000 is necessary for inspection of fiber morphology; this magnification may be obtained by supplementary optical enlargement of the screen image by use of a binocular if it cannot be obtained directly. The TEM shall also be equipped with an energy dispersive X-ray analyzer capable of achieving a resolution better than 175 eV (FWHM) on the MnK_{α} peak. For requirements concerning screen calibration and SAED and ED performance, see Chatfield and Berman (1990);

- a computer system for recording analytical results. As indicated in the section addressing reporting requirements (see Chapter 12), analytical results are to be provided on computer disk (standard, high density 3.5 in or a readable/writeable CD) in a file format that is compatible with EXCEL™. ASCII files are acceptable;

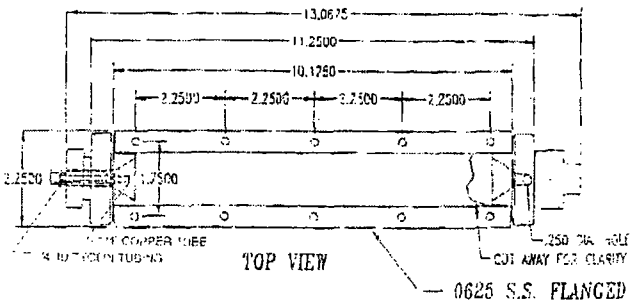
- a laboratory balance capable of mass determination with a resolution of 0.00001 g (1×10^{-5} g) with a capacity of 30 g;

- a laboratory balance capable of mass determination with a resolution of 0.000001 g (1×10^{-6} g) with a capacity of 200 mg;
- a laboratory oven, minimum 1 cu. ft. capacity; and
- a dessicator.

6.4 LABORATORY SUPPLIES

- One No. 8 rubber stopper.
- Two No. 4 rubber stoppers.
- One No. 3 rubber stopper with a three in. length of 1/4 in. copper tubing inserted through it and connected to about 5 ft. of 1/4 in. Tygon tubing.
- Five pounds of $CaNO_3 \cdot 4H_2O$.
- Mixed cellulose ester (MCE) filters: 25 mm dia., 0.47 µm pore size.
- Polycarbonate (PC) filters: 25 mm dia., 0.2 µm pore size.
- Plastic petri dishes for storing 25 mm filters

Figure 6-1: Modification for Exit Hole of Tumbler



Based on Figure A-14 of Berman and Kolk (1997)

FIGURE 6-2:
Elutraitor Bottom Modifications

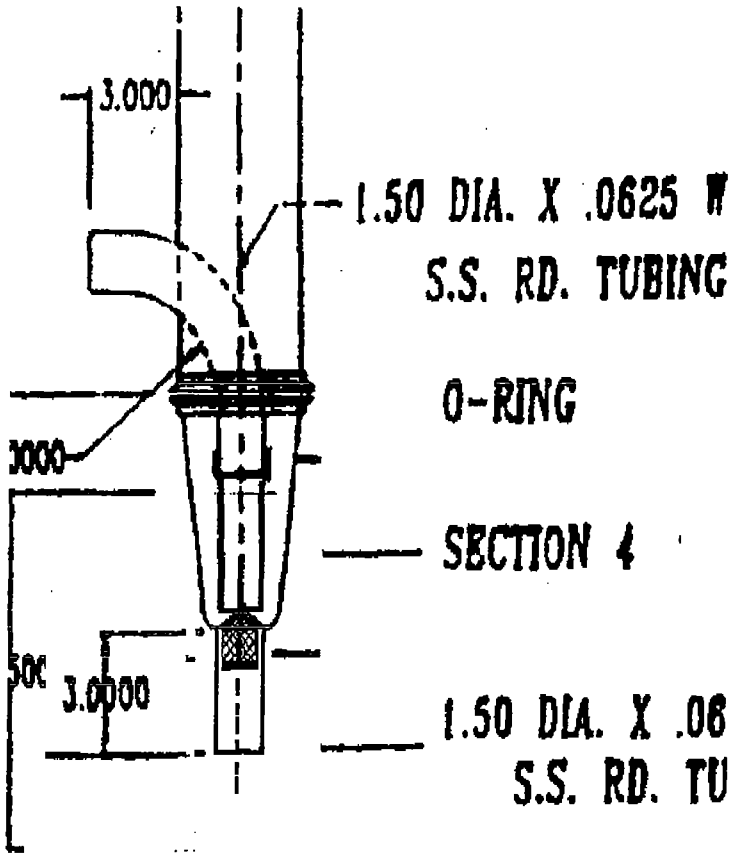
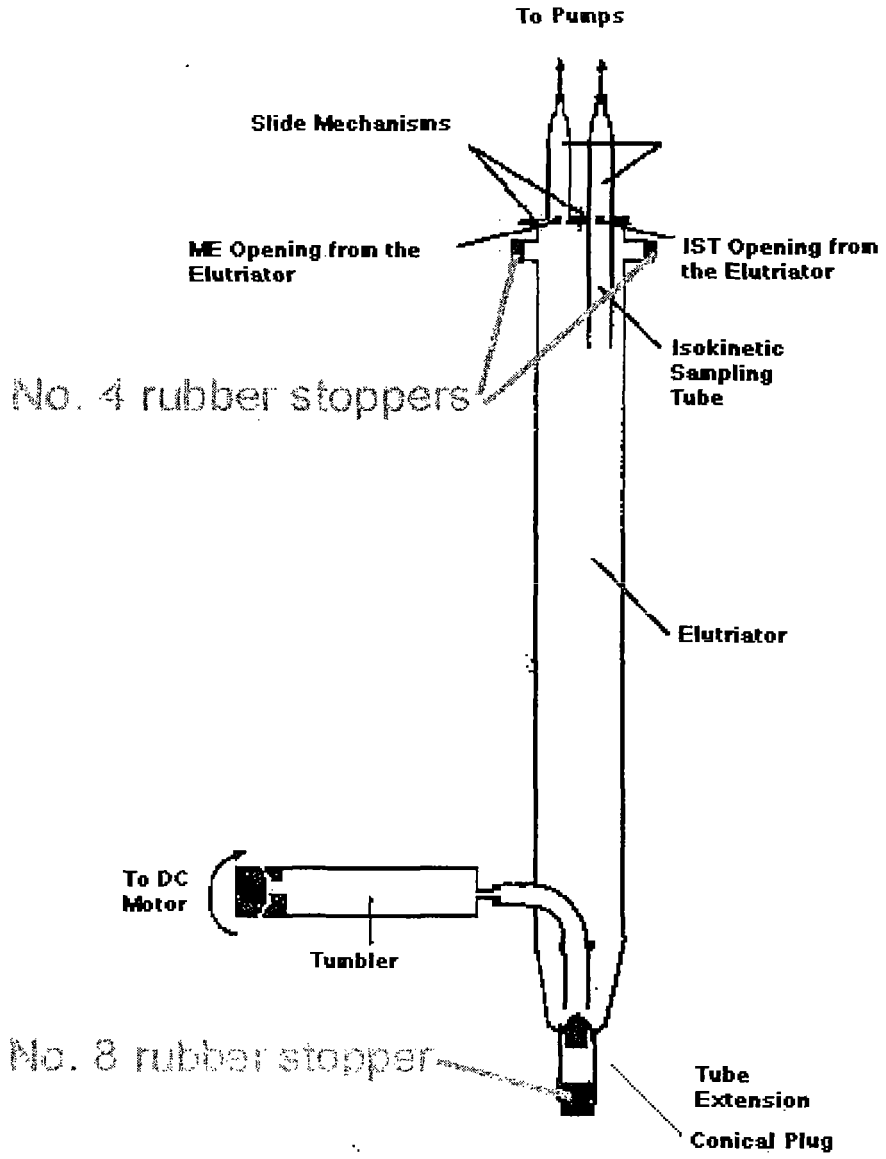


Figure 6-3:
Dust Generator Modifications



From Figure A-1 of Berman and Kalk (1997)

7.0 SOIL OR BULK SAMPLE COLLECTION

Sample collection procedures adopted for this method are flexible to allow adequate sampling of a broad variety of matrices. The method also incorporates several field preparation steps that are designed to preserve sample representativeness while reducing the mass of the samples sent to a laboratory for analysis. Controlling the mass of the samples sent to a laboratory from the field is a cost saving measure; the less material that a laboratory needs to handle as hazardous waste, the lower the total cost of analysis.

WARNING:

MOST OF THE SAMPLE COLLECTION PROCEDURES AND FIELD PREPARATION PROCEDURES DISCUSSED IN THIS DOCUMENT ARE INHERENTLY DUSTY OPERATIONS. THEREFORE, WHEN HANDLING SOILS OR BULK MATERIALS THAT ARE KNOWN TO CONTAIN OR POTENTIALLY CONTAIN ASBESTOS, IT IS IMPERATIVE THAT PROPER RESPIRATORY PROTECTION BE WORN WHILE CONDUCTING THESE PROCEDURES.

As indicated previously (see Section 2.2), measurements derived using this method serve as inputs for emission and dispersion models to predict airborne exposures attendant to disturbance of the sampled matrix. Such models also typically require measurement of the silt content of the sampled matrix. Therefore, it will generally prove prudent to collect at least a subset of samples for simultaneous characterization of the asbestos and silt content of each bulk matrix of interest.

Importantly, however, asbestos concentrations and silt contents serve distinct functions within the emission and dispersion models that are used to predict exposure. For this reason, optimal strategies for sampling a matrix may differ when the goal is to determine asbestos or silt content, respectively. For example, silt content measurements are input into the mathematical terms of these models that reflect activity-specific relationships between the characteristics of the matrix and the magnitude of emissions. It is therefore important that silt content measurements be collected in a manner assuring that they are representative of the effects of the specific release activities being modeled. For example, silt content should be determined for the specific product of a crushing or grinding operation that needs to be modeled.

In contrast, asbestos concentrations determined using this method are expected to reflect an inherent property of each of the materials sampled, which is largely independent of the effects of the release activities of interest (see Section 2.2). Therefore, locations from which soil or bulk samples are to be collected for determination of asbestos concentration shall be selected formally as part of a comprehensive strategy that is designed to provide a representative (unbiased) set of measurements for characterizing the overall mean concentration.

Although further consideration of such issues is required to identify appropriate sampling locations as part of the design of an effective field sampling plan, such considerations are addressed elsewhere (see, for example, Berman and Chesson unpublished) but are beyond the scope of this document. Rather, the remainder of this Chapter focuses on requirements for the collection and handling of individual samples at a site.

7.1 SAMPLE COLLECTION

Any variety of commercially available field sampling equipment (trowels, shovels, augers, corers, etc.) may be used to collect samples for this method. The equipment and procedure(s) selected should be based on the nature of the material being sampled and the depths over which samples are to be collected. Two common examples are presented in the original Superfund Method (Berman and Kolk 1997) so that only a general overview is provided here. Importantly, whatever equipment and procedures are chosen for sampling a particular matrix, they should be applied consistently and invariably at each sampling location within that matrix.

Whatever technique is chosen, the target *minimum* size sample to be collected at each sampling location shall be 1 kg[7]. Larger samples may be required, however, if particularly large (i.e. larger than a 4 or 5 cm in diameter) rocks or debris are present in the material being sampled. To assure representativeness, the largest component sampled should occupy no more than a few percent of the volume of the sample collected (Berman and Kolk 1997). Procedures for compositing samples, if this is desirable, have also been previously presented (Berman and Kolk 1997).

All sampling equipment shall be washed thoroughly with water and detergent between collection of each sample. Sampling equipment shall then be rinsed thoroughly with filtered, distilled water and allowed to air dry. Forced air may be used to expedite drying. If forced air is to be used to facilitate drying, however, such air must be passed through a HEPA filter to prevent delivery of any potential contamination.

Record the identification number, the date, time, and method of collection for each sample in a field notebook. Record the locations from which each sample is collected in the field notebook. Note in the logbook any changes between the sampling locations proposed in the sampling strategy and the actual locations sampled. As indicated previously (Berman and Kolk 1997), such changes are to be avoided to the extent

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[7] It may be impossible to collect kg-size samples for certain types of matrices (e.g. settled dust or manufactured asbestos products) that may be sampled for analysis using this method. The method can handle samples as small as 10 g, although samples smaller than 40 g require special preparation (see Section 8.3). When samples smaller than 1 kg are collected for field preparation and then analysis using this method, the interpretation of results requires special care. For small samples, for example, it is critical to address the consequent limitations that may be associated with compositing or sample homogenization, which may in turn affect representativeness.

possible. If changes are absolutely necessary, clearly document the rationale behind each change.

Supplement written documentation with photographs of each sampling location. This is particularly important if the sampling locations are not laid out on a formal, documented sampling grid that is tied to a permanent field marker.

7.2 FIELD PREPARATION

The specific procedures to be employed for field (and laboratory) preparation will differ depending on the nature of the matrix sampled. Consolidated solids (such as rock samples), for example, will need to be coarse crushed so that the majority of the sample ultimately passes through a 1 cm (3/8 in.) sieve. Such crushing must also be performed in a manner assuring that any fines produced during crushing will be preserved as part of the sample. Alternately, samples that are composed exclusively of fine material to begin with (such as settled dust) can likely be weighed, packaged, labeled, and sent directly to the laboratory without field preparation.

Most field samples will ultimately be handled as unconsolidated, heterogeneous solids. Such materials need to be weighed, separated into a coarse and fine fraction by passing the sample through a 1 cm (3/8 in) sieve, separately weighing the resulting coarse and fine fractions, homogenizing the fine fraction, splitting the fine fraction to produce samples weighing between 40 and 70 g each, and packaging, labeling, and shipping such sample splits to the laboratory for analysis. Detailed procedures for weighing, sieving, homogenizing, splitting, packaging, labeling, and shipment have previously been provided (Berman and Kolk 1997).

8.0 SAMPLE PREPARATION BY DUST GENERATION

The primary purpose for sample preparation by dust generation that is described in this section is to generate dust-laden filters that can be prepared (by direct transfer) for analysis by TEM to determine the concentration of protocol structures (see Chapter 2). The mass of respirable dust, which is deposited on such filters in coincidence with the asbestos, is also determined by weighing the filters. Dust and asbestos measurements are then combined so that asbestos concentrations can be appropriately reported as structures per mass of respirable dust (S/g_{PM10}).

A detailed description of the apparatus employed for dust generation and its theory of operation is provided in Appendix A of Berman and Kolk (1997). Specifications and construction drawings are also provided in that document. Modifications to the original dust generator, which are required to support measurement using this modified elutriator method, are described in Section 6.3 of this document.

8.1 SAMPLE RECEIVING AND STORAGE

All samples received from the field are to be wiped clean with a damp cloth prior to storage or other handling. Samples to be prepared using the dust generator are to be inspected for the presence of free water. If a sample contains free water or if the sample appears visibly moist, it shall be dried at low temperature. Dry the sample in an open, shallow container in an oven that is maintained at a temperature *below* 60° C until the sample comes to constant weight. Note that oven-dried samples may require additional time for conditioning (Section 8.5.2) because the moisture content of the sample will need to be increased to bring it into equilibrium with conditions prevailing in the dust generator.

Once dry, samples between approximately 40 g and 70 g can be loaded directly into the tumbler of the dust generator (Section 8.5.1)[8]. Larger samples must be homogenized and split, as described in Section 8.2, prior to being placed in the tumbler. Smaller samples need to be diluted by mixing with a known mass of washed, play sand prior to loading into the tumbler of the dust generator (see Section 8.3).

8.2 SAMPLE HOMOGENIZATION AND SPLITTING IN THE LABORATORY

Samples received from the field that are larger than approximately 70 g must be dried, as described above, and homogenized and split as described in this section.

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[8] Generally, the denser the sample, the larger the mass that can be tolerated. Ideally, the tumbler should be one-quarter full. Higher volume samples will need to be split prior to loading the tumbler. Lower volume samples will need to be diluted with washed, play sand. The optimum volume for a sample is approximately 36 cm^3.

WARNING:

BECAUSE ASBESTOS CONTAINING DUSTS MAY BE GENERATED FROM THE HANDLING AND PREPARATION OF BULK SAMPLES, ALL OF THE FOLLOWING PREPARATION STEPS SHALL BE PERFORMED IN A PROTECTIVE ENCLOSURE (I.E. A HEPA FILTERED GLOVE BOX OR AN APPROVED FUME HOOD THAT IS DESIGNED TO MINIMIZE EXPOSURE TO LABORATORY PERSONNEL).

As with field homogenization and splitting (described in Section 8.2.3 of Berman and Kolk 1997), either of two options may be selected for homogenization and splitting in the laboratory. When performed in the laboratory, however, such equipment must fit within an appropriately designed, protective enclosure, which is why field preparation may be cost-effective.

Homogenize large volume samples in precisely the same manner as described in Section 8.2.3 of Berman and Kolk (1997). Once samples are homogenized, split samples in precisely the same manner as described in the same section of that document. Continue splitting until a paired set of samples are produced that each contain between approximately 40 g and 70 g of material (or a target volume of 36 cm^3). Record in a laboratory notebook the final masses and identification numbers of the samples that are homogenized and split.

8.3 DILUTING SAMPLES WITH WASHED, PLAY SAND

Samples that are smaller than approximately 40 g shall be diluted with washed, play sand prior to loading in the tumbler.

Weigh out a mass of sand such that the sum of the mass of sand and the mass of the sample of interest will total approximately 70 g. The sand and sample can be mixed directly in the tumbler. Layer half of the sand into the tumbler (in the manner described in Section 8.5.1) so that the material is deposited evenly across the tumbler's bottom. The sample shall then be layered over the sand so that it too is uniformly spread across the sand at the bottom of the tumbler. Finally, layer the rest of the sand over the top of the sample material so that it too is deposited uniformly across the bottom of the tumbler. It is anticipated that the rotation of the tumbler during the initial 1 hour or so of a run, prior to the start of filter collection for asbestos analysis (see Section 8.5.4), will be adequate to homogenize the mix of sand and sample. Alternately, the sand and sample may be thoroughly mixed (by shaking together in a sealed plastic bag) prior to placing the mix in the tumbler. This latter approach may be preferable because it may lessen the time required for dust generation to stabilize (see Section 8.5.4).

> Note, the washed, play sand that is commercially available is generally free of asbestos and contains minimal respirable material. Prior to using such material in this method, however, the sand must be analyzed to confirm its "purity." To analyze the material, place approximately 70 g of candidate sand in the tumbler

of the dust generator. Then, follow all of the steps described in this chapter for generating filters (including sample conditioning and dust generation). As with normal runs, let the dust generator run for about an hour before collecting filters for measurement (Section 8.5.2).

Collect any asbestos (and the associated dust) generated from the sand on filters mounted on the IST opening of the elutriator. Continue passing air through such filters for a period at least *five times* as long as anticipated to be required for loading filters from actual asbestos samples of interest (see Section 8.5.5). The mass of respirable dust deposited on filters over the IST opening of the elutriator from such a run (with the candidate sand) shall then be weighed. Sand can only be used to dilute low volume samples for this method if it exhibits no detectable dust (i.e. less than 5 µg) based on weighing of the filters generated as defined above.

The filters generated from this run with the candidate sand must then be prepared by direct transfer for TEM analysis and analyzed for the determination of asbestos content. Asbestos loading on the sand filters shall be demonstrated to be less than 1 structure in 200 grid openings. Two random samples shall be collected from each 25 kg (50 lb) bag of washed, play sand and analyzed as described above to qualify the sand for use.

Importantly, the largest size particles that can be tolerated on smaller volume samples (to assure adequate homogeneity) are somewhat smaller than the 1 cm target maximum that has been defined for this method. For example, although 1 cm size particles are probably tolerable for samples approaching 40 g total mass, particles no larger than approximately 0.5 cm should be tolerated in samples weighing only 10 g. Field preparation techniques recommended for preparing samples that are shipped to the laboratory for analysis using this method should be adjusted to assure adequate homogenization (that preserves the representativeness of such samples) when the availability of or access to bulk matrices of interest limit the size of samples that can be collected.

8.4 DUST GENERATOR SETUP

Prior to using the dust generator, a supply of at least 15 MCE filters must be conditioned and stored for use. The constant humidity chamber must be loaded with the appropriate solution, and air flow within the dust generator must be calibrated and adjusted.

8.4.1 Conditioning a Stock of Filters

A stock of at least 15 filters (0.45 µm pore size, 25 mm diameter), all from the same filter lot, must be conditioned in a desiccator overnight to bring them into equilibrium with the relative humidity at which they will be used during a run. Place the 15 MCE

filters in a desiccator containing *moist* salt of the same variety as that selected to fill the pans in the humidity control chamber of the dust generator (Section 8.4.2). For most applications, this will be $CaNO_3 \cdot 4H_2O$ (see Appendix A of Berman and Kolk 1997).

After storing the filters overnight in the desiccator, pre-weigh each filter to a minimum precision of ±0.00002 g. Each filter shall then be placed in a separate, covered Petri dish with its weight marked on the top of the container. The lids shall also be numbered sequentially and the filters shall all be used during the run in the order numbered.

Ten polycarbonate filters of either 0.1 or 0.2 µm pore size shall also be weighed on a microbalance with a precision of ±0.000002 g. The polycarbonate filters are not sensitive to humidity changes in the range of 40 to 55% RH. This range can be achieved in an air conditioned office environment. The 0.2 µm pore size is suitable for collecting protocol structures, as required by this method. The 0.1 µm pore size filters are recommended, if fibers as short as 0.5 µm in length are to be collected and counted.

8.4.2 Initiating Humidity Control

Use asbestos-free (filtered, distilled) water to make a 2 L solution of saturated salt. As indicated previously, for most applications, use $CaNO_3 \cdot 4H_2O$, but other salts may be used for specific applications (see Appendix A of Berman and Kolk 1997).

Prepare the solutions by placing 1000 g of the $CaNO_3 \cdot 4H_2O$ into a one L container and adding distilled water to fill the container. The container should be kept capped except when adding additional water or salt on succeeding days, as necessary, until it appears that the solution has stabilized (i.e. no changes in the relative amounts of water and salt are apparent from one day to the next). The solution may also be agitated to accelerate dissolution of the salt and the approach to equilibrium. No water or salt shall be added less than 24 hours from the time that the mixture is to be used, because adequate time is required for the salt solution to come to stable equilibrium.
To load the salt solution, open the top of the humidity control chamber and remove the two pans. Fill each pan with the saturated salt solution being sure that about 25% by volume undissolved salt is also transferred to each pan. Replace the pans and replace the front panel of the plastic enclosure; air should enter the enclosure primarily from the top opening.

8.4.3 Adjusting the Initial Air Flow

The air flow within the various components of the dust generator must be adjusted so that flow within the vertical elutriator will properly separate and pass only respirable particles. Based on the discussion presented in Appendix A (Section A.2.3), the proper linear flow rate in the elutriator shall be set at 0.31 cm/s, which is 5% greater than the Stokes' velocity estimated for the largest spherical, respirable particles (i.e. those with an aerodynamic diameter of 10 µm and unit density).

Next, calculate the required volumetric air flow, V_v, within the elutriator using Equation 8-1:

$$V_v = 81.1 * V_l \qquad (8\text{-}1)$$

where:

V_l is the estimated linear flow rate required to separate respirable particles (i.e. 0.31 cm/s); and

V_v is the corresponding volumetric flow rate (cm^3/s) through the elutriator.

The coefficient, "81.1", in Equation 8-1 corresponds to the cross-sectional area of the elutriator (in cm^2) with the internal diameter given in the drawings supplied in the Superfund Method (Berman and Kolk 1997)[9].

> Note: the following discussion, which describes procedures both for adjusting air flow within the dust generator and for checking for leaks, assumes familiarity with the component pieces of the dust generator. It is therefore recommended that the reader familiarize themselves with the operation of the dust generator by reading Chapter 9 and Appendix A of Berman and Kolk (1997), prior to proceeding with the rest of this section.

8.4.3.1 Determining proper air flow for a dust generation run. Using Equation 8-1, V_v is calculated to be 25.14 cm^3/s. This is the volumetric flow rate that must be maintained in the elutriator to prepare filters suitable for analysis by this method. This flow must then be divided between flow out of the ME opening of the elutriator and the IST opening of the elutriator.

Given the specifications indicated in the construction drawings for the elutriator (Figures A-15 and A-17 in Appendix A of Berman and Kolk 1997), the cross-sectional area of the isokinetic sampling tube is 4.7% of the total internal cross-section of the elutriator. Therefore, during a run, flow must be adjusted so that 4.7% of the flow in the elutriator (1.19 cm^3/s or 72 ml/min) passes through the IST opening of the elutriator and the remaining 95.3% (23.9 cm^3/s or 1,430 ml/min) passes through the ME opening.

> Note, if the design of the elutriator being used for this method differs from that defined in Berman and Kolk 1997 (and modified as described of Section 6.3 of this document), air flow requirements will have to be adjusted accordingly.

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[9] If the design of the dust generator being used for this method differs from the that defined in Berman and Kolk 1997, Equation 8-1 will need to be adjusted accordingly.

To prepare for a run, the elutriator must first be properly set up. Filter cassettes should be attached to the flowmeters and the pumps of the dust generator through the stopcocks as shown in Figure 8-1[10]. Appropriate filters must then be mounted in each of the four cassettes: 0.45 µm MCE filters (over backing pads) are mounted in the filter cassettes over the ME opening of the elutriator and 0.2 µm polycarbonate filters (with 0. 45 µm MCE filters as backing pads) are mounted in each filter cassette of the IST filter assembly.[11]

A FILTER CASSETTE CONTAINING A 0.45 µm MCE FILTER AND BACKING PAD IS ALWAYS PLACED BETWEEN EACH PRECISION FLOWMETER AND ITS CORRESPONDING PUMP FOR SAFETY REASONS. THIS IS TO PREVENT ASBESTOS FIBERS FROM BEING PUMPED INTO LABORATORY AIR SHOULD ONE OF THE FILTER CASSETTES ON THE DUST GENERATOR FAIL OR SHOULD SOMEONE INADVERTANTLY FORGET TO MOUNT A FILTER INTO ONE OF THE SLIDER CASSETTES DURING OPERATION.

When the elutriator is being run, each stopcock is positioned so that the active filter on each slider is the only one connected to its pump. Just before an active filter is switched, stopcock(s) are adjusted so that flow is now directed through both the active and the inactive filter on a slider. The slider is then repositioned to place the new filter over the opening of the elutriator. Stopcock(s) are then adjusted again so that flow to the old (previously active filter) is shut off and all of the flow is now directed through the new, active filer.

> Note: when a slider is being repositioned with both stopcocks open (or a corresponding, three-way stopcock adjusted to allow flow to both filters), the flow out of the elutriator is never completely shut off because the space between the two filter mounts on the slider is less than the diameter of the hole into the elutriator.

Once the elutriator is properly configured, the flow meters calibrated, and the tumbler loaded (Section 8.5.1), the dust generator is ready for use. Turn on the pumps and adjust flow so that the required 1,430 ml/min pass through the ME opening of the elutriator (with a loaded filter mounted over the opening) and the required 72 ml/min pass through the IST opening and the associated filter cassette[12]. Check for variation

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[10] One modification to the setup depicted in Figure 8-1 that has proven helpful is to replace each assembly of multiple stopcocks and glass "Y" tubes depicted in the figure with single, three-way stopcocks.

[11] The indicated configuration is appropriate for measurement of protocol structures, which are all longer than 5 µm. If there is a need to count shorter structures, use 0.1 µm pore size PC filters in the cassettes over the IST opening of the elutriator.

[12] It has been observed that increasing the flow through the IST opening increases the efficiency of deposition on filters mounted over this opening. If necessary to facilitate collection, flow through the IST

in flow as sliders and valves are adjusted (as when "changing out" filters) during a run and correct accordingly. Flow through the ME opening should vary by no more than ± 10% from the required value as flow is switched between the two filter mounts on the associated slider. Flow through the IST opening should vary by no more than -0% to +20% as flow is switched between the two filter mounts on the associated slider. Importantly, the tolerance range is adjusted upward for the IST opening so that the *minimum* acceptable volume flow rate is the target value. It is, however, acceptable, for air flow in the IST opening to slightly exceed the target value.

8.4.3.2 Checking for leaks. The filter assembly over the IST opening of the elutriator shall first be checked for leaks prior to assembling the dust generator for a run because the low flow rate in this filter assembly (i.e. 72 ml/min) makes it very sensitive to small leaks. Similar leaks do not affect the performance of the filter assembly over the ME opening of the elutriator because flow in this assembly is so much higher under normal operating conditions.

To check the integrity of the filter assembly over the IST opening, remove the top section off the elutriator and insert the No. 3 stopper with the Tygon tubing into the bottom of the isokinetic tube. Place a backing pad and filter into each cassette on the slider. Next open the stopcock(s) that connect one filter of this assembly to the entrance end of one of the 375 ml flowmeters. The exit end of this flowmeter (which is the flowmeter that will read air flow through the IST opening during actual dust generator runs) is then connected through a filter cassette to a pump.

> **A FILTER CASSETTE CONTAINING A 0.45 µm MCE FILTER AND BACKING PAD IS ALWAYS PLACED BETWEEN THE FINAL FLOWMETER AND THE PUMP FOR SAFETY REASONS. THIS IS TO PREVENT ASBESTOS FIBERS FROM BEING PUMPED INTO LABORATORY AIR SHOULD ONE OF THE FILTER CASSETTES ON THE DUST GENERATOR FAIL OR SHOULD SOMEONE INADVERTANTLY FORGET TO MOUNT A FILTER INTO ONE OF THE SLIDER CASSETTES DURING OPERATION.**

Position the filter with the open stopcock(s) so that it is aligned directly over the IST opening on the elutriator top. Connect the tubing from the stopper at the bottom of the isokinetic sampling tube to the exit end of another 375 ml flowmeter, the entrance to which is left open to the air.

When the pump connected to the filter assembly in the manner described above is turned on, the flow path is from the air, through the first flowmeter, into the bottom of the isokinetic sampling tube, through the IST opening at the top of the elutriator, through the active filter cassette, through the second flowmeter (which is the flowmeter that will read air flow during actual dust generator runs), through the safety filter cassette, and to the pump.

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opening can be increased by as much as 70% over its nominal value. This has virtually no effect on the overall operation of the elutriator.

Turn the pump on and adjust the flow through the filter to the 72 ml/min level on the second flowmeter (i.e. the one that will be used to monitor flow during actual runs). The first flowmeter should also read the same flow within 7%. If the difference is greater than 7%, the system shall be checked for leaks. When both flowmeters read 72 ml/min plus or minus 7%, the stopcocks should be switched so that flow now occurs through both filters on the slider and the readings again compared. Both should still read 72 ml/min ± 7%.. The slider shall then be positioned with the second filter over the IST opening and the stopcock(s) adjusted to direct flow only through the second filter. The readings on the two flowmeters should remain the same, within the stated tolerance.

It is prudent also to check for leaks in the assembly over the ME opening of the elutriator. To accomplish this, remount the isokinetic tube onto the opening for the ME filters and repeat the above-described procedure at the same *low* flow rate to check for leaks in the filter assembly over the ME opening. If no leaks are detected in that slider assembly, the isokinetic tube is returned to its original position (under the IST opening) in preparation for a run.

Once leak detection is complete, the elutriator can be reassembled in preparation for a run.

8.5 DUST GENERATOR OPERATION

To prepare asbestos samples using the dust generator: load the tumbler, condition the bulk sample, wait for dust generation to stabilize, and collect appropriately loaded filters for asbestos analysis by TEM following preparation by direct transfer.

8.5.1 Loading the Tumbler

Detach the tumbler from its drive motor and the vertical elutriator and remove it from the plastic enclosure at the bottom of the dust generator (see Appendix A of Berman and Kolk 1997). Place the tumbler on a support surface and open the top for loading. Be sure that the tumbler is clean prior to loading.

Introduce a sample by holding the sample container against the inner lip of the tumbler and tilting the container so that the sample pours smoothly into the tumbler. Move the sample container back and forth along the length of the tumbler to facilitate uniform deposition of the sample in the tumbler. When pouring is complete, tap the sample container vigorously so that the quantitative transfer is complete. The masses of samples introduced into the tumbler shall range between 40 g and 70 g. Larger samples shall be homogenized and split prior to loading as described in Section 8.2. Samples smaller than 40 g can also be run by first diluting them with washed, play sand (Section 8.3).

Shake the tumbler gently to assure uniform deposition of the sample within the tumbler, which should be no more than about one quarter full. Be sure that the rubber gasket on the tumbler is in good repair and properly seated. Replace the gasket if it is worn. Secure the top of the tumbler with 10 screws and replace the tumbler within the plastic enclosure at the bottom of the dust generator. Reattach the elutriator entrance tube and D.C. motor to the tumbler (see Appendix A of Berman and Kolk 1997).

8.5.2 Conditioning the Sample

Before conditioning the sample, be sure that the dust generator has been properly set up. This means, check that:

- the pans in the constant humidity chamber have been filled with saturated salt solution;
- appropriate size and type filters have been mounted in each of the four cassette mounts on the slide mechanisms atop the elutriator;
- air flow valves have been properly set; and
- all air lines between the dust generator, flow valves, and pumps are properly configured (see Section A.1.1 of Appendix A of Berman and Kolk 1997).

To condition the sample, turn on all pumps and begin the flow of air through the dust generator. ***DO NOT TURN ON THE TUMBLER MOTOR.*** Allow the flow of air to continue for a minimum of two hours before beginning a run.

8.5.3 Initiating a Run

Once the sample has been conditioned. set the tumbler drive motor to 30 rpm and turn it on. Simultaneously, move the two slide mechanisms at the top of the elutriator so that new, clean filters are now aligned over both the ME and IST openings of the elutriator. Be sure to change the valve orientations on the lines leading to the filters so that air flow is directed through both filter cassettes of each slider while the sliders are moved and then to the filter cassettes that are newly aligned with the elutriator openings.

Replace the filters originally aligned over the elutriator openings (but no longer aligned) with clean filters and weigh and store the old filters in labeled Petri dishes. These filters are conditioning filters and may be useful for isolating a problem should corrective actions be required.

8.5.4 Monitoring to Confirm Stable Dust Generation

The rate of respirable dust generation is monitored at the start of a sample run to indicate when conditions have stabilized[13]. Filters to be used for determination asbestos concentration shall only be collected after dust generation has stabilized. Dust generation is monitored by recording the deposited mass on each of a set of filters that are sequentially changed out of the filter mounts over the ME opening of the elutriator at defined, regular intervals.

Initially, change out the filters aligned over the ME opening of the elutriator at intervals of between ten and twenty minutes. The change is accomplished by moving the slide mechanism to switch a new filter into alignment at the same time that the old filter is switched out of alignment. Prior to sliding the filters, be sure to adjust the stopcock(s) so that flow is simultaneously directed to both the active filter (i.e. the one over the opening to the elutriator) and the inactive filter (i.e. the one that is out of the line of air flow).

After sliding the new filter into alignment, turn the associated stopcock(s) so that the air flow is directed only through the new filter. Dismount and replace the old filter (the one previously exposed to the air stream from the elutriator) with a fresh filter and place the exposed filter in the Petri dish in which it was initially stored after the determination of its tare mass. Repeat this process at regular intervals to generate a series of filters that represent dust sequentially collected from the air flow in the generator. If required to better understand this process for monitoring dust generation, a more detailed description is provided in Section 9.4.4 of Berman and Kolk (1997).

Along with the proper identifier, record the times during which air flow is started and halted for each filter. Weigh each filter after dismounting. Record the initial and final masses of the filter and the net mass of dust deposited on the filter (i.e. the difference between the initial tare mass and the final mass). Determine the net amount of time during which dust was being deposited on the filter by subtracting the start time from the stop time (in seconds). Then compute the average rate at which dust was deposited on the filter by finding the ratio of the net mass deposited to the net time over which deposition occurred.

After collecting dust on the first two or three filters, the interval over which dust is collected on each filter may be optimized. The ideal mass of dust to be deposited on each filter is between 0.002 and 0.006 g. Estimate the interval of time required to deposit approximately 0.006 g and exchange later filters at the optimum rate. When adjusting the time for dust deposition, however, be sure to avoid depositing more than 0.006 g of dust on any filter because the probability that a portion of the deposit will be lost during handling increases as the mass of the deposit on the filter increases.

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[13] Dust generation can also be monitored and modeled for extended periods, if there is a desire either to estimate the absolute concentration of respirable dust in the original sample or to explore the dynamics of dust generation for the particular matrix being analyzed (see Berman and Kolk 1997 for details).

Once dynamic equilibrium is achieved, so that dust generation stabilizes, the rate of release of dust from a sample in the tumbler has been shown to decrease slowly and regularly with time as the reservoir of dust within the sample is depleted. Such decay has been shown to follow first-order kinetics (see Section A.1 of the Appendix).

That dust generation has stabilized will be apparent when the computed rate of dust deposition on each subsequent filter that is collected over the ME opening of the elutriator indicates a slow, monotonic decrease with time[14]. Rate estimates for individual filters should not vary from their nominal value (predicted from a linear trend) by more than 5%. When this pattern is observed, filters can then be collected over the IST opening of the elutriator for determination of the concentration of asbestos (Section 8.5.5). Typically, about an hour of run time is required before conditions stabilize sufficiently to allow collection of filters for asbestos analysis.

8.5.5 Collecting Filters for Asbestos Analysis

In preparation for collecting filters for asbestos analysis, estimate the time that will be required for optimal deposition. Estimate the optimum time required to collect dust on the filters to be used for asbestos analysis using Equation 8-2.

$$t_{opt} = 20*M_{dep}/D_{rate} \qquad (8\text{-}2)$$

where:

t_{opt} is the time required to collect a filter deposit of mass M_{dep} (s);

M_{dep} is the target mass of the deposit on the filter (g); and

D_{rate} is the estimated rate of deposition for the specific run over the time interval of interest (g/s).

The derivation of Equation 8-2 can be found in Section A.2.2 of the Appendix. Equation 8-2 is simply a recapitulation of Equation A-10, with the terms redefined.

Recent experience with this method (Kolk unpublished) indicates that the optimum loading for filters to be prepared for asbestos analysis using this method is approximately 100 µg of respirable dust. By substituting this value for M_{dep}, Equation 8-2 can be further simplified:

$$t_{opt} = 0.002/D_{rate} \qquad (8\text{-}3)$$

where all terms have been previously defined.

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[14] In some cases the rate of decrease is so small that the rate of dust deposition appears steady.

The D_{rate} in Equation 8-3 is estimated simply from the deposition rates estimated from the last set of filters collected over the ME opening of the elutriator. t_{opt} can then be derived accordingly. To expedite asbestos measurement using this method, it is recommended (but not required) that a set of multiple filters be collected, which bracket the optimum collection time estimated using Equation 8-3. After superficial examination of gird specimens prepared from such filters, the optimally loaded filter can then be selected for final analysis.

> Note: to collect optimally loaded filters over the IST opening, the flow rate through the opening may be increased by as much as 70% over its nominal value. Although this will have no impact on elutriator performance, dust deposition over the IST opening has been found to be very sensitive to minor changes in this flow rate.

Dust is collected on filters mounted over the IST opening in the same manner previously described for filters mounted over the ME opening of the dust generator. Polycarbonate (PC) filters shall be used for collecting asbestos samples because PC filter masses have been found to be less affected by humidity changes than MCE filters (Kolk unpublished). Filters should be left in line with the air flow over the IST opening for the period of time approximately equal to t_{opt} (estimated using Equation 8-3).

Following dust collection, each PC filter shall be weighed on the microbalance to determine whether the target dust mass was successfully collected. Unfortunately, a substantial electrostatic charge builds up on these filter during deposition and this charge must be neutralized before an accurate weight can be obtained.

Cahn, a manufacturer of microbalances, sells radioactive sources that can be placed in the weighing chamber and also sells solutions of radioactive salts that can be used to coat the inside of the weighing chamber. There is a technical note available from Cahn indicating how to minimize the effects of electrostatic charge. Care must be taken when eliminating static charges to assure that particles are not loss from the filter surface. Another way to eliminate charges on a filter is to place the filter in a covered Petri dish lined with a conducting material that is ground and that is maintained at a high relative humidity, but not sufficiently high to cause condensation. The filter is left for several hours to allow the charge to dissipate. Although this latter method is slower than using a radioactive source to neutralize charge, it has been found to be less problematic (with much lower potential for disturbing the dust deposit on the filter). It is this latter procedure that was used during method tests and found to be satisfactory.

It is recommended that approximately five filters be collected for asbestos analysis during each run with the loading on each such filter increased sequentially by increasing deposition time. If there appears to be difficulty obtaining adequate deposits on these filters, the air flow through the isokinetic sampling tube can be increased by as much as 70% beyond nominal to facilitate particle collection. This will have virtually no impact on the elutriator's ability to separate respirable and non-

respirable particles. The filter chosen for preparation and TEM analysis shall be the filter that exhibits a deposited mass closest to 100 µg.

After the five filters are collected, the run may be terminated. However, it is recommended that the dust generator assembly be left undisturbed until the PC filters are determined to be satisfactory for TEM examination.

When the static charge has dissipated to the extent that it does not interfere with weighing, the PC filters shall be weighed. If one or more of the filters is within 10% of the target weight of deposit (100 µg), the tumbler can be emptied, the remaining material archived, and the elutriator cleaned.

8.6 CLEANING THE DUST GENERATOR

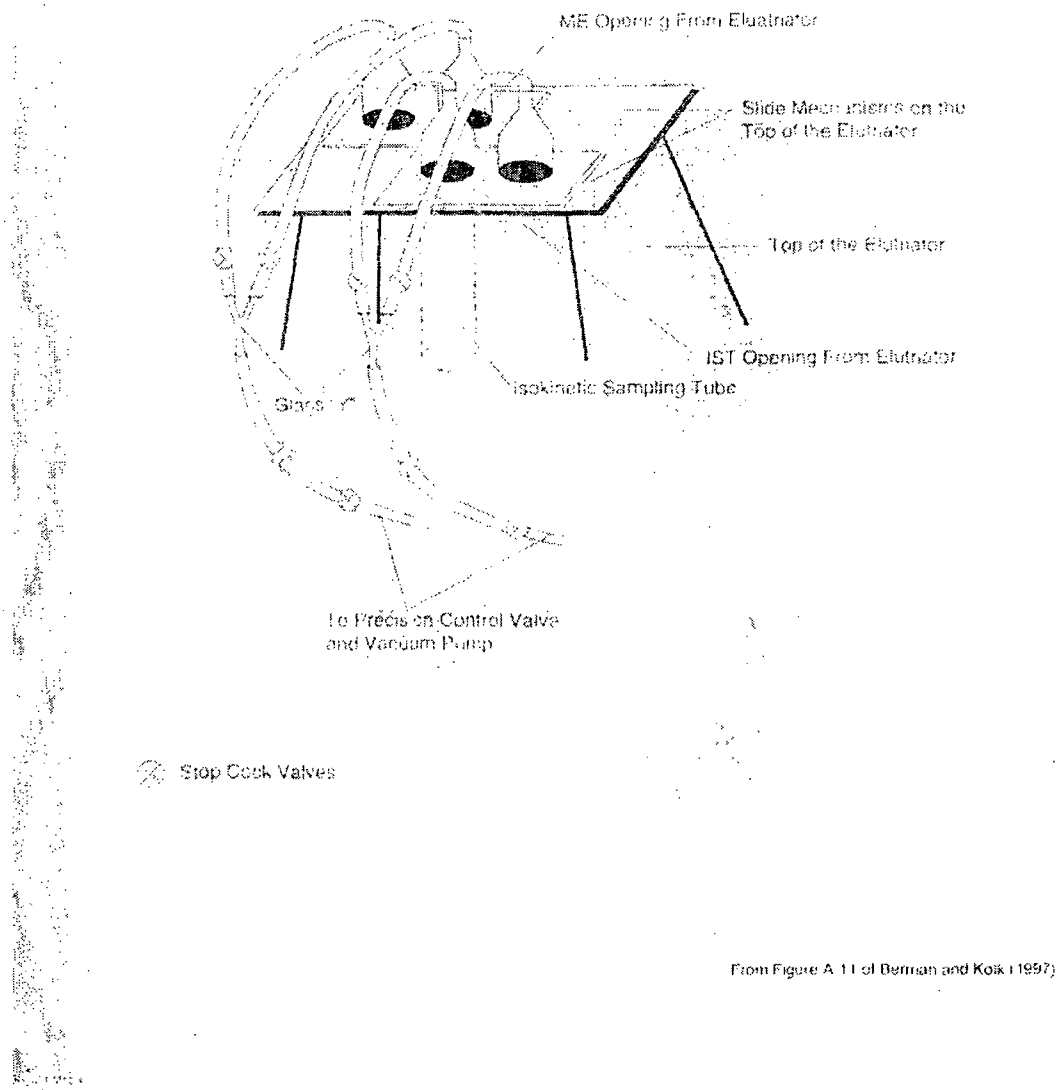
8.6.1 Cleaning the Elutriator

The dust generator is designed for quick and easy assembly and disassembly to facilitate cleaning. Most of the joints are simple friction couplings or ring clamp couplings. To clean the dust generator, carefully disconnect and disassemble the tumbler, remove the plug and dust collector system from the elutriator, decouple the sections of the elutriator tube, disassemble the slide mechanisms of the dust collector. Be careful to not disturb or spill any of the dust on the bottom and sides of the elutriator. Conduct the transfer of all material left in the elutriator to a container that can be capped. The metal pieces of the dust generator may then be washed with biodegradable detergent and rinsed with asbestos-free water. The pieces may then be left to dry in room air or may be dried with a forced, HEPA-filtered air stream. Discard asbestos containing waste according to applicable regulations.

8.6.2 Optional Method for Disassembling the Elutriator

An optional cleaning procedure for soils that are believed to be heavily contaminated with respirable asbestos is as follows. Use a 1/2 gallon size pressurized tank garden spray to wet the inside of the elutriator. The ME filter holder is removed to expose the opening at the top of the elutriator and the mist spray is injected into the opening until the contents of the elutriator are lightly wetted. The spray is also applied to the connection to the tumbler while detaching the tumbler from the elutriator. The spray is then directed into the tube leading into the bottom of the elutriator and the inside lightly misted. The elutriator may then be disassembled and cleaned. The contents of the tumbler can be archived and the soil removed from within the elutriator can also be dried and archived if desired. Again any contaminated waste should be given proper disposal.

Figure 8-1:
Tubing Connections for Filter Cassettes Mounted On the Elutriator



From Figure A-11 of Demian and Kolk (1997)

9.0 PREPARATION OF SPECIMEN GRIDS FOR TEM ANALYSIS

Prepare speciment grids from filters collected over the IST opening of the elutriator.

Although this method specifies that filters collected over the IST opening of the elutriator shall be prepared using a direct transfer technique, an indirect transfer technique is also described, as an option for certain applications. The primary purpose for considering indirect transfer is that it may prove cost-effective under special circumstances. Importantly, however, one must also consider that concentration measurements derived from indirectly prepared samples are not generally good predictors of risk (see Berman and Crump 1999a). Thus, indirect preparation should be used in association with this method only after appropriate consideration of the consequences.

9.1 SPECIMEN GRID PREPARATION USING A DIRECT TRANSFER TECHNIQUE

Filters collected over the IST opening of the elutriator (as described in Section 8.5.5) shall be prepared using the direct transfer technique that is described in Section 10.5 of the ISO Method (ISO 10312). The loading calculation described in Section 8.5.5 Equation 8-3) is for a filter loading of 100 µg. If a different loading is desired, the appropriate target mass needs to be substituted into the equation to estimate the appropriate time for collection. A loading of over 150 µg is not recommended.

From each filter that has been collected over the IST opening, prepare four specimen grids from locations on the filter that are each separated by 90° radially and also prepare one grid from the center of the filter. The four outer grids should be about 75% of the distance from the center to the active edge of the filter (i.e.about 8.3 mm from the center of the filter). Thus, a total of 5 grid specimens shall be prepared from each filter.

9.2 SPECIMEN GRID PREPARATION USING AN INDIRECT TRANSFER TECHNIQUE

As an option to the procedure described in Section 9.1 above (for special applications *only),* filters collected over the IST opening of the elutriator (as described in Section 8.5.5) may also be prepared using the indirect transfer technique that is described in Sections 10.3 to 10.5 of the ISO Method (ISO 10312). For this option, multiple sections of the most highly loaded filter obtained from the dust generator shall be prepared using a range of dilutions to allow selection of the optimally loaded specimen grids for final, detailed analysis.

Alternately the more highly loaded filters collected over the ME opening of the elutriator (to monitor the stability of dust generation as described in Section 8.5.4) may also be prepared by indirect transfer for asbestos analysis.

10.0 PROCEDURES FOR ASBESTOS AND DUST ANALYSIS

10.1 PROCEDURES FOR ASBESTOS ANALYSIS

Specimen grids prepared as described in Chapter 9 are to be analyzed using transmission electron microscopy (TEM). Follow the procedures for analysis described in the ISO Method (ISO 10312) including procedures for:

- examining specimen grids to determine acceptability for analysis;
- structure counting by TEM (except that counts are limited to parent structures and components that qualify as protocol structures and the determination of the stopping point is modified);
- structure morphological classification;
- structure mineralogical identification; and
- blank and quality control determinations.

The stopping points for the analyses conducted in support of this method are a function of the required sensitivity for the method and are defined in Section 10.1.2 below.

Begin by examining one of each set of specimen grids derived for a defined loading from a particular run of the dust generator and select the optimally loaded set for analysis. Use the criteria for determining the acceptability of specimen grids (from the ISO Method) to define optimal loading.

When performing detailed analysis, be sure to distribute asbestos counts evenly over the entire set of specimen grids prepared from a particular filter at a defined (optimal) loading. Record the morphology and mineral type of asbestos structures as described in the ISO Method (ISO 10312). Importantly, because protocol structures are limited to structures longer than 5 µm, scans for counts of protocol structures can be performed at a magnification of 10,000x, which should expedite analysis relative to higher magnification scans.

10.1.1 Counting Rules for Asbestos

Because this method is focused specifically on deriving counts of asbestos protocol structures, count only structures longer than 5 µm that are also thinner than 0.5 µm. Qualifying structures that are longer than 10 µm also need to be distinguished for separate enumeration. Include in the count all isolated structures that exhibit the required dimensions and identifiable components of complex structures that also exhibit qualifying morphology. Chrysotile and amphibole structures must be separately enumerated. For amphiboles, identify the specific mineral type.

10.1.2 Analysis of Specimen Grids Prepared from Filters Collected Over the IST Opening of the Elutriator

Prior to initiating a detailed analysis of specimen grids, the stopping rules for the analysis must be defined. Assuming these specimen grids have been prepared using a direct transfer technique (as discussed in Section 9.1), define the stopping rules for the detailed analysis as follows.

First, calculate the maximum number of grid openings that will have to be scanned during the analysis from the relationship:

$$N_{go} = S_d * A_f * / (S_{smpl} * A_{go} * \Delta M_f) \qquad (10\text{-}1)$$

where:

N_{go} is the maximum number of grid openings to be scanned;

S_d is the number of structures required to define detection using the analysis (defined here as 1);

A_f is the total area of the filter from which the specimen grids were prepared (mm^2). A typical value for the surface area of a filter is 385 mm^2;

S_{smpl} is the required analytical sensitivity for the method (s/g_{PM10}). A target analytical sensitivity of $3x10^6$ s/g_{PM10} is defined in Section 2.4, which should be adequate for most studies ;

A_{go} is the area of a single grid opening (mm^2). A typical value for the area of a grid opening is 0.01 mm^2; and

ΔM_f is the mass of respirable dust collected on the filter from which the specimen grids were prepared (g). As indicated in Section 8.5.5, a reasonable target loading is $1x10^{-4}$ g.

Given a target sensitivity of $3x10^6$ s/g_{PM10} and typical values for each of the parameters of Equation 10-1 (listed above), approximately 150 grid openings will need to be counted for a typical sample. Importantly, to count protocol structures, such scans need to be performed at a magnification of only 10,000x.

The number of grid openings to be scanned for specific analyses shall be determined by substituting case-specific values for the above listed parameters into Equation 10-1.

Stop the counting, characterization, identification, and recording of asbestos structures on a particular analysis when one of the following obtains:

- the scan is completed for the grid opening on which the 25th asbestos protocol structure that is longer than 10 µm is counted; or
- a sufficient number of grid openings are scanned to achieve the target analytical sensitivity.

Note, because a total of 5 grid specimens are to be prepared from each filter, it is practical to modify the above-listed stopping rules so that they can be applied to each of the individual grid specimens from a particular filter. Thus, stop counting, characterization, identification, and recording of asbestos structures on a particular grid specimen when:

- the scan is completed for the gird opening on which the 5th asbestos protocol structure that is longer than 10 µm is observed on the particular grid specimen being scanned; or

- one fifth of the total number of grid openings required to achieve the target analytical sensitivity are scanned on the particular grid specimen.

The above rules must then be applied to each of the five grid specimens that are prepared from a particular filter.

10.2 EVALUATING THE RATE OF RELEASE OF RESPIRABLE DUST

10.2.1 Determining the Mass of Respirable Dust on Filters to be Prepared for Asbestos Analysis

To support the determination of asbestos concentrations using this method, it is necessary to determine the mass of dust that is simultaneously deposited on the filter. This is because, as indicated in Section 2.2, asbestos concentrations are to be reported as the ratio of asbestos to dust (S/g_{PM10}).

For filters collected over the IST opening of the elutriator (or filters collected over the ME opening, if these are to be used for determination of asbestos concentrations using the optional indirect transfer technique described in Section 9.2), determine the dust mass on the filter by weighing the filter directly. As indicated in Section 8.5.5, a microbalance is required to determine the net weight of the PC filters collected over the IST opening because the mass of dust on such filters will not be more than 150 µg.

10.2.2 Optional Procedure for Determining the Mass of Respirable Dust in a Bulk Sample

Although not required in this modified elutriator method, an optional procedure is presented here for determining the mass of respirable material in bulk materials that are analyzed for asbestos content using this method. This procedure is modified from the one originally reported in Section 11.2 of the Berman and Kolk (1997) to account for the modifications to the configuration of the dust generator that are incorporated into this method.

10.2.2.1 Evaluating the rate of release of respirable dust. The rate of release of respirable dust from a sample prepared using the dust generator is estimated from measurements of the mass of dust collected over time on the set of filters mounted over the ME opening of the elutriator. The measurements used specifically are from those filters that are collected while the tumbler is operating at the *highest* rotation rate employed for the sample (see Section A.2.1 of Appendix A).

Begin by plotting the cumulative mass collected on the filters as a function of time. To derive the cumulative mass for a particular time interval, add the mass of dust measured on the filter collected from that time interval to the sum of the masses measured on the set of filters collected earlier in the run. Typical curves are depicted in Figures 11-1 and 11-2 of Berman and Kolk (1997). Next, calculate the cumulative mass released from the sample over time from the cumulative mass collected on filters over time using the relationship developed in Section A.2.1 of Appendix A:

$$M_r = 1.0523 * M_f \qquad (10\text{-}2)$$

where:

M_r is the cumulative mass of dust released from a sample between the start of a run and time "t" (g); and

M_f is the cumulative mass collected on filters between the start of a run and time "t" (g).

Equation 10-2 is appropriate to use to relate the mass of dust collected on filters to the mass released from the sample when air flow in the dust generator is setup as indicated in Section 8.4.3. If different air flow conditions are established for a particular experiment, the relationship between M_r and M_f will have to be derived using Equation A-4 from Appendix A.

The total mass of dust in the sample at the beginning of the run must next be estimated using the relationship developed in Appendix A. Based on the relationship (see Section A.2.1):

$$\ln(M_o - M_r) = \ln(M_o) - kt \qquad (10\text{-}3)$$

where:

M_o is the mass of dust in the sample at the start of the run (g);

k is the first-order rate constant for the release of dust from the sample (s^{-1}); and

t is the time since the start of the run (s).

A plot of $\ln(M_o - M_r)$ versus t should be a straight line with a slope equal to the rate constant for the release of dust from the sample and an intercept equal to the natural logarithm of the mass of dust in the sample at the start of the run. Derive estimates of "M_o" and "k" by programming Equation 10-3 into a spreadsheet and running a regression[15].

Input a range of guesses for the value of M_o into the spreadsheet and run a regression to fit a value for k and to calculate a value for the regression coefficient, "r^2" for each value of M_o. Plot the regression coefficient, "r^2" as a function of M_o. An example of such a plot is presented in Figure 11-3 of Berman and Kolk (1997). The value of M_o that provides the fit with the largest regression coefficient (i.e. with r^2 closest to 1) shall be reported as the correct value for the mass of dust in the sample at the start of the run and shall be reported with the corresponding k value as the estimated rate constant for dust release from the sample during the run.

10.2.2.2 Determining the content of respirable dust. To determine the mass percent of respirable dust in the original sample, first determine the total mass of respirable dust in the sample *at the start of a run* for the last run completed on the sample, which is derived as described in the last section.

If the dust generator run analyzed as described in Section 10.2.2 was preceded by a run with the tumbler speed set at 30 rpm, which will generally be the case when the goal is to determine the mass of respirable dust in the bulk sample (see Section 9.4.4 of Berman and Kolk 1997), to estimate the total mass of dust present in the sample, it is necessary to sum the masses collected on the ME filters during all of the earlier runs

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[15] Any of several commercial spreadsheet programs (including, for example, EXCEL™ or LOTUS™) contain the necessary capabilities and may be employed to derive optimum values for "M_o" and "k."

from the same sample, call this M_E. Then adjust this mass for the additional mass of dust that would simultaneously have passed through the IST opening of the elutriator:

$$M_{OE} = 1.053*M_E \qquad (10\text{-}4)$$

where:

M_{OE} is the cumulative mass of respirable dust released from a sample during all runs completed prior to the current run (g); and

M_E is the cumulative mass of respirable dust measured on filters collected over the ME opening during all runs completed prior to the current run (g).

Calculate the total mass of dust originally present in the sample, M_{tot}, by summing the mass released during all earlier run(s) with the mas of dust estimated to have resided in the sample at the beginning of the final (usually higher rpm) run, M_o. M_o is equal to the mass of respirable dust remaining in the sample at the end of the earlier run(s). M_o will have been derived as described in Section 10.2.2.1:

$$M_{tot} = M_{rOE} + M_o \qquad (10\text{-}5)$$

where all parameters have been previously defined.

Finally, estimate the mass percent of respirable dust in the bulk sample as follows:

$$\%RD = 100*M_{tot}/M_{sample} \qquad (10\text{-}6)$$

where:

%RD is the mass percent of respirable dust in the sample (%); and

M_{sample} is the mass of the original sample placed in the tumbler (g).

10.3 DETERMINING THE CONTENT OF ASBESTOS

As previously indicated (Section 2.2), the asbestos concentrations determined using this method are reported as the ratio of asbestos to dust (S/g_{PM10}). Also as previously indicated (Section 2.1), concentrations of protocol structures longer than 10 µm must be separately enumerated from structures between 5 and 10 µm in length. Concentrations for these two size categories shall be reported separately.

Whatever length (or other size) category of structures is to be determined, calculate concentrations as follows:

$$C_{dust} = S_c * A_f / (N_{go} * A_{go} * \Delta M_f) \qquad (10\text{-}7)$$

where:

C_{dust} is the concentration of asbestos per unit mass of respirable dust (S/g_{PM10});

S_c is the number of structures (of a defined size and type) that are counted during the scan (#);

A_f is the area of the filter on which the asbestos (and dust) were deposited for analysis (mm^2);

N_{go} is the total number of grid openings scanned during the analysis (#);

A_{go} is the area of a grid opening (mm^2); and

ΔM_f is the mass of respirable dust deposited on the filter.

11.0 QUALITY ASSURANCE QUALITY CONTROL REQUIREMENTS

The quality assurance/quality control (QA/QC) requirements indicated in the ISO Method (ISO 10312) shall be considered relevant and appropriate when using this method. In addition, the following blank and duplicate/replicate schedule shall be employed when running samples using this method.

11.1 BLANKS

The following blanks shall be collected routinely in concert with use of this method:

- *lot blanks or filter blanks.* Two filters from each lot of 50 filters obtained from the manufacturer shall be prepared using a direct transfer procedure and analyzed to assure that background contamination on the filters does not exceed 0.2 S/mm^2. Only filters from lots whose blanks pass the defined criterion shall be used in support of this method;

- *laboratory blanks.* A sufficient number of laboratory blanks shall be collected, prepared using a direct transfer technique and analyzed to show that the room in which bulk samples are handled and prepared satisfy the requirements defined in Section 10.6 of Chatfield and Berman (1990). When laboratory blanks indicate that room air is out of compliance with the stated criterion, use of this method is to cease until appropriate corrective actions are completed;

- *field blanks.* Field blanks shall be collected during any sample collection activities performed in association with use of this method. The number of such blanks to be collected and the schedule for their analysis shall be determined based on the complexity of the anticipated sampling scheme and shall be defined as part of the sampling plan for the site. QC criteria for field blanks will also be set as part of the planning for the study;

- *method blanks.* Method blanks should be collected and analyzed at a minimum frequency of one for every 20 samples analyzed using this method. The cumulative loading of asbestos structures observed on method blanks must not exceed 0.2 S/mm^2. Note that it may prove advantageous to collect method blanks more frequently than one every 20 samples. The additional blanks can then be stored and analyzed at a later date, if needed to troubleshoot for corrective action;

- *equipment blanks.* Equipment blanks and method blanks can be considered to be interchangeable. However, method blanks offer the advantage of simultaneously evaluating the laboratory's supply of washed sand. As long as no problem exists with the washed sand, run method blanks in lieu of running equipment blanks. However, should a problem develop with the washed sand, equipment blanks can be substituted for method blanks with no loss of method

performance (other than preventing the analysis of small volume samples for lack of appropriately qualified washed sand); and

- *conditioning filters.* Conditioning filters are collected at the beginning of every run. These filters do not need to be prepared and analyzed routinely but should be stored because they may prove useful for troubleshooting should corrective actions be required.

11.2 DUPLICATES AND REPLICATES

A fixed fraction (5 to 10%) of the samples collected in the field in support of this method shall be collected as spatial duplicates (two samples collected at immediately adjacent locations or two composites composed of an independent, inter-located set of samples representing the same area or volume). These shall be labeled and sent to the laboratory in such a manner so as to assure that laboratory personnel cannot identify them as duplicates. The frequency of collection of spatial duplicates shall be defined as part of the sampling plan for the site. Comparison of the results of the analysis of such samples provides a measure of all of the components of total precision except population variability.

As indicated previously (Section 8.2.1), 100% of the samples shipped from the field are to be shipped as duplicate pairs. The laboratory shall randomly select 2 or 3% of the duplicate samples shipped from the field and shall analyze both samples of the pairs so selected. Comparison of the results of the analysis of such samples, which are homogenized splits of the same sample, provides an indication of the precision achieved by sample preparation and analysis.

Should analysis of duplicate pairs indicate an unacceptable degree of variability (i.e. a relative percent difference greater than 50%), replicate counts shall be performed on designated samples by multiple analysts in the laboratory (or by the same analyst on different days). Laboratory management shall assign such counts so as to assure that analysts cannot determine which counts are replicates. Results of such replicate counts shall serve to distinguish whether the major source of variability observed among duplicate pairs is due to analysis or to sample preparation. Appropriate corrective actions may then be devised.

11.3 INTER-LABORATORY PERFORMANCE PROGRAMS

For large projects, it is highly recommended that analyses be performed by multiple laboratories (at least two) and that a well-crafted, formal inter-laboratory proficiency program be instituted to gauge the relative performance of the laboratories. In addition to the standard components of such a program (such as laboratory audits and consensus standards), such a program should include blind field replicates sent occasionally for comparison both between laboratories and within each laboratory.

12.0 REPORTING REQUIREMENTS

This chapter indicates field and laboratory reporting requirements.

12.1 FIELD AND LABORATORY NOTEBOOKS

Over the course of the project, information critical to the proper reporting and interpretation of each sample analysis will be developed both in the field and in the laboratory. Formal procedures are required to preserve such information and to allow for the documentation of attendant information that, while not employed directly in the calculation of results, may provide insight into the interpretation of such results.

Details of the reporting requirements for field and laboratory notebooks are provided in Section 13.1 of Berman and Kolk (1997).

12.2 FIELD ACTIVITIES REPORT

To assure that the field information required to complete estimation of dust and asbestos concentrations and release rates are provided to the data users, a field activities report must be completed and must be submitted to the laboratory along with the corresponding samples. Laboratory personnel are then to attach this report directly to their batch report, which shall cover the corresponding batch of samples.

Details of the reporting requirements for field activities reports are provided in Section 13.2 of Berman and Kolk (1997).

12.3 SAMPLE ANALYSIS REPORT

The sample analysis report for each sample shall include the following information, at a minimum:

(a) reference to this method;

(b) reference to the sample identification and batch number for the sample;

(c) the date and site from which the sample was collected;

(d) the weights and identities of the coarse and fine fractions of the sample and the sub-sample of the fine fraction sent for analysis;

(e) the weights and identities of any splits or other fractions of the sample generated during laboratory preparation;

(f) the weight and identity of the sub-sample placed in the dust generator;

(g) the relative flow rates through the IST and ME openings of the elutriator during each run of the dust generator for the sample;

(h) the measured mass of respirable material on each filter that is collected over the IST opening (or ME Opening) of the elutriator and prepared for asbestos analysis;

(I) if other than protocol structures, a statement identifying the structure size and type categories of interest for the study (see Chatfield 1993);

(j) a statement of the minimum acceptable identification category and the maximum identification category attempted for asbestos structures counted and characterized during analysis (see Chatfield 1993);

(k) a statement specifying which identification categories and which structure categories have been included in the counts of structures employed to estimate concentrations (see Chatfield 1993);

(l) the counts of asbestos protocol structures (separately, for total protocol structures and protocol structures longer than 10 µm and separately for chrysotile and amphibole structures);

(m) the active area of the filter prepared for asbestos analysis along with the average area of grid openings on specimen grids prepared from each filter and the number of grid openings scanned during analysis;

(n) the estimated concentration of asbestos protocol structures (separately, for total protocol structures and protocol structures longer than 10 µm and separately for chrysotile and amphibole structures). Concentrations are to be reported as S/g_{PM10};

(o) the analytical sensitivity achieved for a particular analysis; and

(p) 95% confidence limits for the concentrations reported.

An example of the format to be employed for a sample analysis report is presented in Figure 12-1.

FIGURE 12-1
SAMPLE ANALYSIS REPORT FORMAT

Laboratory Name
Laboratory Address
Laboratory Contact
Telephone Number

Report Date
Project Name (Optional)

METHODS:
(reference this method)

Date Analysis Started (M/D/Yr)
Date Analysis Completed (M/D/Yr)
Analyst(s) Initials

Laboratory Sample No.
Field Sub-Sample Identification No.
Field Preparation Technique (**Attach a Copy of the Relevant Field Activities Report**)
Additional Laboratory Preparation Procedures (*describe any employed*)
Sample Drying
Sample Splitting
Other

TEM Analysis:
Effective Area of Analytical Filter (sq mm)
(*Indicate whether from ME or from IST Opening*)
Magnification
Grid Opening Area (sq mm)
Number of G.O. Scanned
Asbestos Struc;ture Size and Type Categories of Interest (*see Chaffield 1993*)
Minimum Acceptable Structure Identification Category (*see Chaffield 1993*)

Dust Generator
Mass of Sample Tumbled (g)
Air Flow Rate Through ME Opening of Dust Generator (ml/min)
Air Flow Rate Through IST Opening of Dust Generator (ml/min)
Estimated Total Air Flow Rate Through Elutriator (ml/min)

Filters from the Isokinetic Sampling Tube (IST) Opening of the Elutriator
Mass of respirable dust on filter (g)

FIGURE 12-1
SAMPLE ANALYSIS REPORT FORMAT (Cont.)

Laboratory Name — Report Date

Laboratory Sample No.

	No. of Protocol Structures	
	Total	Long (> 10 µm)
Asbestos Analysis Results[16]:		
No. of Chrysotile Asbestos Structures	XXX	XXX
No. of Amphibole Asbestos Structures	XXX	XXX
(Indicate Amphibole Mineral Type)		

ESTIMATED ASBESTOS CONCENTRATIONS (S/g_{PM10})

	Concentrations	
	Mean	95% UCL
Total Chrysotile Protocol Structures	XXX	XXX
Long Chrysotile Protocol Structures	XXX	XXX
Total Amphibole Protocol Structures	XXX	XXX
Long Amphibole Protocol Structures	XXX	XXX
Total Asbestos Protocol Structures	XXX	XXX
Long Asbestos Protocol Structures	XXX	XXX
Estimated Analytical Sensitivity: *(structures/g_{PM10})*	*XXX*	*XXX*

(Attach a Copy ot the TEM Raw Data Sheets)

[16] Protocol structure counts shall include all qualifying parent structures and qualifying components of complex structures (enumerated per the counting rules of the ISO Method)

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APPENDIX A:
CONSTRUCTION AND OPERATION OF A DUST GENERATOR/ ELUTRIATOR FOR ISOLATING AND CONCENTRATING RESPIRABLE DUST AND ASBESTOS FROM SOILS AND OTHER BULK MATERIALS OF INTEREST

The dust generator/elutriator incorporated into this Modified Elutriator Method for the Determination of Asbestos in Soils and Bulk Materials is designed to isolate and concentrate respirable material and asbestos that is present within soils or bulk materials of interest. A detailed description of the device (including specifications and construction drawings) is provided in Berman and Kolk (1997) along with a discussion of the theory of operation for the device. Changes to the design of the original device that are required to support this modified method are described in Section 6.3. A brief description of the motivation for each of the changes is provided below, followed by a refined and abbreviated discussion of the theory of operation of the new device.

A.1 DUST GENERATOR MODIFICATIONS

A.1.1 Elimination of the Scrubber

The purpose of the scrubber in the initial design was to provide a slurry of respirable particles from which filters could be prepared for TEM examination using an indirect transfer procedure. Since it has now been shown that TEM analysis of samples prepared by direct transfer best correlate with risk (Berman and Crump 1999a), this modified method incorporates TEM specimen grid preparation by direct transfer, which obviates the need for the scrubber. In fact, even though an optional indirect transfer procedure is incorporated into this modified method, the preferred alternative is to use filters collected over the ME opening of the elutriator as the source of material for indirectly prepared samples, rather than rely on a scrubber. Thus, the scrubber has been removed from the modified design for the device (see Figure 6-3).

A.1.2 Elimination of the Secondary Air Source

In the original configuration of the device, a side inlet to the cup at the bottom of the elutriator was left open as a secondary source of air. This initially allowed air flow within the tumbler to be varied independent of flow in the elutriator. It was also thought that a source of air providing upward flow at the bottom of the elutriator would minimize the chance that respirable particles would be lost from the air stream from the tumbler by passing through quiescent sections of the flow regime at the bottom of the elutriator.

It was later discovered, however, that the laminar flow regime within the elutriator did not allow for adequate mixing of the two air streams (the one from the tumbler and the one from auxiliary entrance at the bottom of the elutriator). This was not a problem when TEM filters were being prepared by indirect transfer from scrubber material. However, it creates non-uniform deposits on filters collected over the IST opening of the elutriator, which limits their utility for analysis following direct transfer. Therefore,

the auxiliary opening at the bottom of the elutriator is sealed in the modified design. Moreover, the detailed design of the bottom of the elutriator has also been modified (see Section 6.3) to assure that flow velocities between the tumbler and the elutriator never fall below the minimum velocity in the main body of the elutriator. This assures that respirable material is not inadvertently lost, even in the absence of the auxiliary flow of air.

A.1.3 Reduction in the Size of the Exit Hole from the Tumbler

When the tumbler operates for a few hours with air constantly flowing in one direction, it was found that there is gradual movement of the mass of the material in the tumbler toward the exit hole. This is expected because, as the particles in the tumbler are tossed into the air stream, they are always pushed toward the exit, regardless of their size. Eventually, the level of the material in the tumbler at the end near the exit hole would rise to the point where some of the material would be forced out through the exit hole. This problem was minimized by reducing the diameter of the exit hole and extending the tube that serves as the exit hole a small distance into the tumbler so that it is no longer flush with the wall of the tumbler (see Figure 6-1).

A.1.4 Modifications to Humidity Control

As indicated in Berman and Kolk (1997), optimum generation of dust occurs at a relative humidity of approximately 50%. In the original method, the salt solution recommended for maintaining constant humidity contains potassium carbonate dihydrate, which actually provides control at a relative humidity of 43% at 20° C. Calcium nitrate tetrahydrate is recommended in the current method because, at equilibrium with a saturated solution, humidity is maintained at a value very close to 50%. It turns out that control of relative humidity that is closer to the ideal is more critical for this modified method to help mitigate the effects of static charging, which are much more pronounced on the required polycarbonate (PC) filters than on MCE filters. As indicated in Section 8.5.5, static electricity must be controlled to minimize problems during weighing of the PC filters on the microbalance.

A.2 THEORY OF OPERATION

A.2.1 The Dynamics of Dust Generation

The dynamics of the release of dust from a sample during a run using the dust generator have been evaluated so that the rate of release and mass of dust in the sample can be derived from measurements of the mass of dust deposited over time on the set of filters collected over the ME opening of the elutriator. Analysis of data obtained from several different types of samples during the pilot study for this method (Berman et al.1994) indicate that the rate of release of mass from a sample in the dust generator is well described by a first-order rate equation:

$$-dM_s/dt = k*M_s \qquad \text{(A-1)}$$

where:

M_s is the mass of respirable dust remaining in the sample at time "t" (g);

t is the time since the start of the run (s); and

k is the first-order rate constant for the release (s^{-1}).

The minus sign in this equation indicates that mass is lost with time.

Equation A-1 can be integrated to yield:

$$\ln(M_s) = \ln(M_o) - kt \qquad \text{(A-2)}$$

where:

M_o is the mass of respirable dust in the sample at the start of the run (i.e. at time t = 0) (g).

Given that "M_s" can also be expressed as the difference between "M_o" and "M_r" the cumulative mass released up to time "t," Equation A-2 can also be expressed as:

$$\ln(M_o - M_r) = \ln(M_o) - kt \qquad \text{(A-3)}$$

where:

M_r is the cumulative mass released between the start of a run and time "t" (g).

The relationship presented in Equation A-3 indicates that a plot of the natural logarithm of the quantity (M_o -M_r) versus time should be a straight line with a slope equal to the rate constant for dust release, k, and an intercept equal to the initial mass of dust in the sample at the start of the run, M_o. The cumulative mass of dust released from the sample over time, M_r, can be derived from measurements of dust collected on filters during the run. However, because M_o also appears as part of one of the parameters that must be plotted to evaluate the relationship expressed in Equation A-3, the value of M_o must be optimized using regression, as described in Section 10.2.2.1 of the main text of this method.

The cumulative mass released from a sample at time "t" during a run, "M_r" is directly proportional to the cumulative mass measured on filters collected during the run:

$$M_r = M_f*(F_d + F_c)/F_c \qquad (A\text{-}4)$$

where:

M_f is the cumulative mass measured on filters collected from filters mounted over the top of the elutriator up to time "t" (g);

F_d is the percent of airflow through the IST opening of the elutriator (%); and

F_c is the percent of airflow through the ME opening of the elutriator (%).

Because F_d plus F_c must sum to unity and F_d will typically have been set at 4.75% during the initial setup of the dust generator[17] (see Section 8.4.3.1), Equation A-4 reduces to:

$$M_r = 1.05*M_f \qquad (A\text{-}5)$$

As indicated above, values for M_o must be derived by performing a regression analysis of the relationship described by Equation A-3. This can be accomplished by using any of several commercially available spreadsheet programs (such as, for example, EXCEL™ or LOTUS™). The procedure to be followed to derive estimates of M_o and k are described in Section 10.2.2.1.

A.2.2 The Time Dependence of Dust Collection

As indicated in Section A.2.1 above, the generation of dust from the tumbler is well described by the first order rate equation:

$$-dM_s/dt = k*M_s \qquad (A\text{-}1)$$

However, experience gained during the pilot study for this method (Berman et al. 1994) further indicates that the rate of change of M_s is sufficiently slow in most cases such that, for periods of no more than 20 minutes, M_s can be considered constant. Thus, for estimating such things as the time required to load individual filters in the dust generator, a simpler form of Equation A-1 can be used (in which M_s is considered constant):

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[17] This assumes that the design specifications for the elutriator being used by the laboratory conform to those defined in Berman and Kolk 1997.

$$\Delta M_s = k^* M_s^* \Delta t = k' \Delta t \qquad \text{(A-6)}$$

where:

M_s is still the mass of respirable dust remaining in the sample at time "t" but it is assumed constant over the short interval of time "Δt" (g);

ΔM_s is the mass of respirable dust released from the sample over the short time interval "Δt" (g);

Δt is a relatively short time interval (no more than 20 minutes) during which the release of dust is being estimated (s);

k is still the first-order rate constant for the release (s^{-1}); and

k' is an empirically derived, linear rate constant that is appropriate for any particular, short time interval (s^{-1}).

Due to the geometry of the dust generator, the mass of respirable dust deposited on a filter in the dust generator in any short time interval, call this ΔM_f, is simply the product of the dust released from the sample, ΔM_s, and the fraction of the flow through the dust generator that is also directed through that filter. Thus, for filters collected over the isokinetic sampling tube (the IST opening of the elutriator):

$$\Delta M_{f\ (IST)} = 0.047^* \Delta M_s = 0.047^* k'^* \Delta t \qquad \text{(A-7)}$$

Similarly, for the ME opening over the elutriator:

$$\Delta M_{f(ME)} = 0.953^* \Delta M_s = 0.953^* k'^* \Delta t \qquad \text{(A-8)}$$

Comparison of Equations A-7 and A-8 shows that the k' constants for the two equations must be identical. Moreover, it is possible to estimate k' (without knowledge of M_s) from dust mass measurements made on filters collected over one opening of the elutriator, which can then be extrapolated to estimate the amount of time required to collect a defined mass of dust on filters collected over the other opening of the elutriator. Thus, by solving both Equations A-7 and A-8 for k' and substituting one into the other, the following relationship obtains:

$$\frac{\Delta M_{f(ME)}}{0.953 \Delta t} = \frac{\Delta M_{f(IST)}}{0.047 \Delta t} \qquad \text{(A-9)}$$

Combining terms and re-arranging yields the following relationship for determining the time required to collect a defined mass on a filter over the IST opening of the elutriator, given an estimated deposition rate that is defined based on dust mass measurements made on filters collected over the ME opening of the elutriator:

$$\frac{20 * \Delta M_{f(IST)}}{\left(\frac{\Delta M_{f(ME)}}{\Delta t}\right)} = \Delta t \qquad \text{(A-10)}$$

Where:

$\Delta M_{f(IST)}$ is a target mass of dust to be deposited on a filter mounted over the IST opening of the elutriator (g);

Δt is the time required to deposit a mass of dust equal to $\Delta M_{f(IST)}$ on a filter mounted over the IST opening of the elutriator (s);

$\left(\frac{\Delta M_{f(ME)}}{\Delta t}\right)$ is the rate of deposition of dust on a filter estimated as the ratio of the net dust deposited on a filter mounted over the ME opening of the elutriator to the net time required to deposit the observed mass (g/s).

A.2.3 Size Separation Using the Vertical Elutriator

The operation of the vertical elutriator has been described in detail in Section A.2.3 of the original Superfund Method (Berman and Kolk 1997).

Designation: C 702 – 98 (Reapproved 2003)

American Association State Highway and Transportation Officials Standard: T 248

Standard Practice for Reducing Samples of Aggregate to Testing Size[1]

This standard is issued under the fixed designation C 702; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers three methods for the reduction of large samples of aggregate to the appropriate size for testing employing techniques that are intended to minimize variations in measured characteristics between the test samples so selected and the large sample.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 125 Terminology Relating to Concrete and Concrete Aggregates[2]

C 128 Test Method for Density, Relative Density (Specific Gravity), and Absorption of Fine Aggregate[2]

D 75 Practice for Sampling Aggregates[3]

3. Terminology

3.1 *Definitions*—The terms used in this practice are defined in Terminology C 125.

4. Significance and Use

4.1 Specifications for aggregates require sampling portions of the material for testing. Other factors being equal, larger samples will tend to be more representative of the total supply. This practice provides procedures for reducing the large sample obtained in the field or produced in the laboratory to a convenient size for conducting a number of tests to describe the material and measure its quality in a manner that the smaller test sample portion is most likely to be a representation of the larger sample, and thus of the total supply. Failure to carefully follow the procedures in this practice could result in providing a nonrepresentative sample to be used in subsequent testing. The individual test methods provide for minimum amount of material to be tested.

4.2 Under certain circumstances, reduction in size of the large sample prior to testing is not recommended. Substantial differences between the selected test samples sometimes cannot be avoided, as for example, in the case of an aggregate having relatively few large size particles in the sample. The laws of chance dictate that these few particles may be unequally distributed among the reduced size test samples. Similarly, if the test sample is being examined for certain contaminants occurring as a few discrete fragments in only small percentages, caution should be used in interpreting results from the reduced size test sample. Chance inclusion or exclusion of only one or two particles in the selected test sample may importantly influence interpretation of the characteristics of the original sample. In these cases, the entire original sample should be tested.

5. Selection of Method

5.1 *Fine Aggregate*—Reduce the size of samples of fine aggregate that are drier than the saturated-surface-dry condition (Note 1) using a mechanical splitter according to Method A. Reduce the size of samples having free moisture on the particle surfaces by quartering according to Method B, or by treating as a miniature stockpile as described in Method C.

5.1.1 If the use of Method B or Method C is desired, and the sample does not have free moisture on the particle surfaces, moisten the sample to obtain free moisture on the particle surfaces, mix thoroughly, and then reduce the sample size.

5.1.2 If use of Method A is desired and the sample has free moisture on the particle surfaces, dry the entire sample to at least the saturated-surface-dry condition, using temperatures that do not exceed those specified for any of the tests contemplated, and then reduce the sample size. Alternatively, if the moist sample is very large, make a preliminary split using a mechanical splitter having chute openings of 38 mm (1½ in.) or more in width to reduce the sample to not less than 5000 g. Dry the portion so obtained, and reduce it to test sample size using Method A.

NOTE 1—The method of determining the saturated-surface-dry condition is described in Test Method C 128. As a quick approximation, if the

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[1] This practice is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.20 on Normal Weight Aggregates.

Current edition approved May 10, 1998. Published December 1998. Originally approved in 1971. Last previous edition approved in 1993 as C 702 – 93.

[2] *Annual Book of ASTM Standards*, Vol 04.02.

[3] *Annual Book of ASTM Standards*, Vol 04.03.

fine aggregate will retain its shape when molded in the hand, it may be considered to be wetter than saturated-surface-dry.

5.2 *Coarse Aggregates and Mixtures of Coarse and Fine Aggregates*—Reduce the sample using a mechanical splitter in accordance with Method A (preferred method) or by quartering in accordance with Method B. The miniature stockpile Method C is not permitted for coarse aggregates or mixtures of coarse and fine aggregates.

6. Sampling

6.1 Obtain samples of aggregate in the field in accordance with Practice D 75, or as required by individual test methods. When tests for sieve analysis only are contemplated, the size of the field sample listed in Practice D 75 is usually adequate. When additional tests are to be conducted, the user shall be satisfied that the initial size of the field sample is adequate to accomplish all intended tests. Use similar procedures for aggregate produced in the laboratory.

METHOD A—MECHANICAL SPLITTER

7. Apparatus

7.1 *Sample Splitter*—Sample splitters shall have an even number of equal width chutes, but not less than a total of eight for coarse aggregate, or twelve for fine aggregate, which discharge alternately to each side of the splitter. For coarse aggregate and mixed aggregate, the minimum width of the individual chutes shall be approximately 50 % larger than the largest particles in the sample to be split (Note 2). For dry fine aggregate in which the entire sample will pass the 9.5-mm (⅜-in.) seive, a splitter having chutes 12.5 to 20 mm (½ to ¾ in.) wide shall be used. The splitter shall be equipped with two receptacles to hold the two halves of the sample following splitting. It shall also be equipped with a hopper or straight-edged pan which has a width equal to or slightly less than the over-all width of the assembly of chutes, by which the sample may be fed at a controlled rate to the chutes. The splitter and accessory equipment shall be so designed that the sample will flow smoothly without restriction or loss of material (see Fig. 1 and Fig. 2).

NOTE 2—Mechanical splitters are commonly available in sizes adequate for coarse aggregate having the largest particle not over 37.5 mm (1½ in.).

8. Procedure

8.1 Place the original sample in the hopper or pan and uniformly distribute it from edge to edge, so that when it is introduced into the chutes, approximately equal amounts will flow through each chute. Introduce the sample at a rate so as to allow it to flow freely through the chutes and into the receptacles below. Reintroduce the portion of the sample in one of the receptacles into the splitter as many times as necessary to reduce the sample to the size specified for the intended test. Reserve the portion of material collected in the other receptacle for reduction in size for other tests, when required.

METHOD B—QUARTERING

9. Apparatus

9.1 Apparatus shall consist of a straight-edged scoop, shovel, or trowel; a broom or brush; and a canvas blanket approximately 2 by 2.5 m (6 by 8 ft).

10. Procedure

10.1 Use either the procedure described in 10.1.1 or 10.1.2 or a combination of both.

10.1.1 Place the original sample on a hard, clean, level surface where there will be neither loss of material nor the accidental addition of foreign material. Mix the material thoroughly by turning the entire sample over three times. With the last turning, shovel the entire sample into a conical pile by depositing each shovelful on top of the preceding one. Carefully flatten the conical pile to a uniform thickness and diameter by pressing down the apex with a shovel so that each

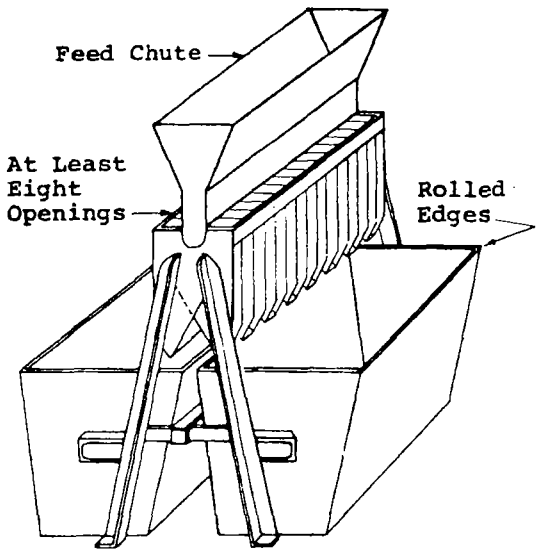
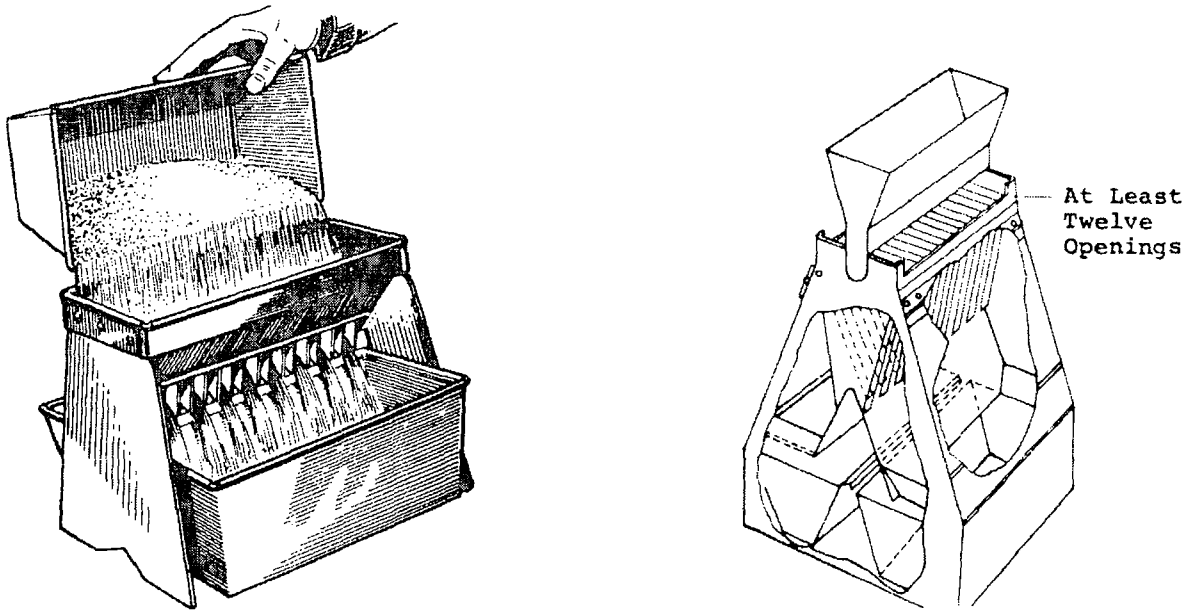


FIG. 1 Large Sample Splitter for Coarse Aggregate



NOTE— Small Sample Splitters for Fine Aggregate. May be constructed as either closed or open type. Closed type is preferred.

FIG. 2 Sample Splitters (Riffles)

quarter sector of the resulting pile will contain the material originally in it. The diameter should be approximately four to eight times the thickness. Divide the flattened mass into four equal quarters with a shovel or trowel and remove two diagonally opposite quarters, including all fine material, and brush the cleared spaces clean. Successively mix and quarter the remaining material until the sample is reduced to the desired size (Fig. 3).

10.1.2 As an alternative to the procedure described in 10.1.1, when the floor surface is uneven, place the field sample on a canvas blanket and mix with a shovel as described in 10.1.1, or by alternately lifting each corner of the canvas and pulling it over the sample toward the diagonally opposite corner causing the material to be rolled. Flatten the pile as described in 10.1.1. Divide the sample as described in 10.1.1, or if the surface beneath the blanket is uneven, insert a stick or pipe beneath the blanket and under the center of the pile, then lift both ends of the stick, dividing the sample into two equal parts. Remove the stick leaving a fold of the blanket between the divided portions. Insert the stick under the center of the pile at right angles to the first division and again lift both ends of the stick, dividing the sample into four equal parts. Remove two diagonally opposite quarters, being careful to clean the fines from the blanket. Successively mix and quarter the remaining material until the sample is reduced to the desired size (Fig. 4).

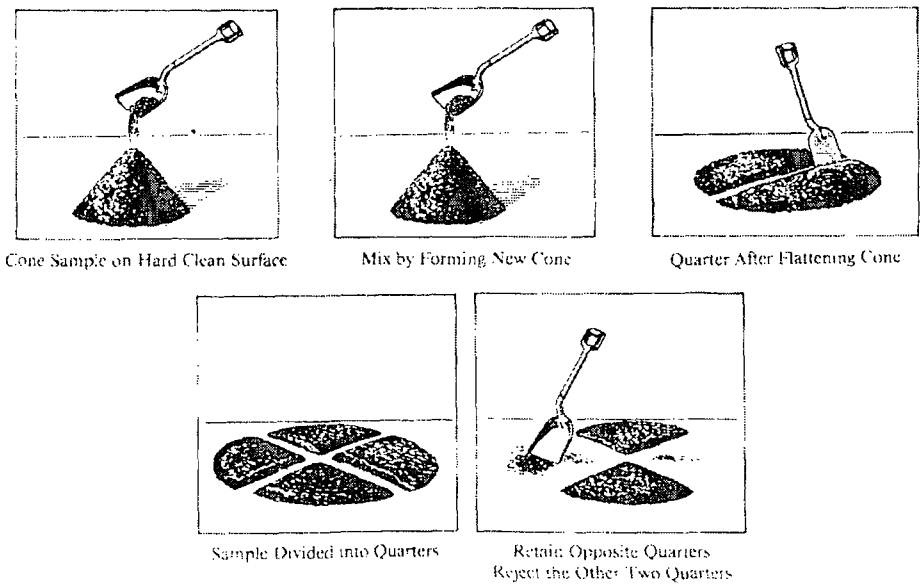


FIG. 3 Quartering on a Hard, Clean Level Surface

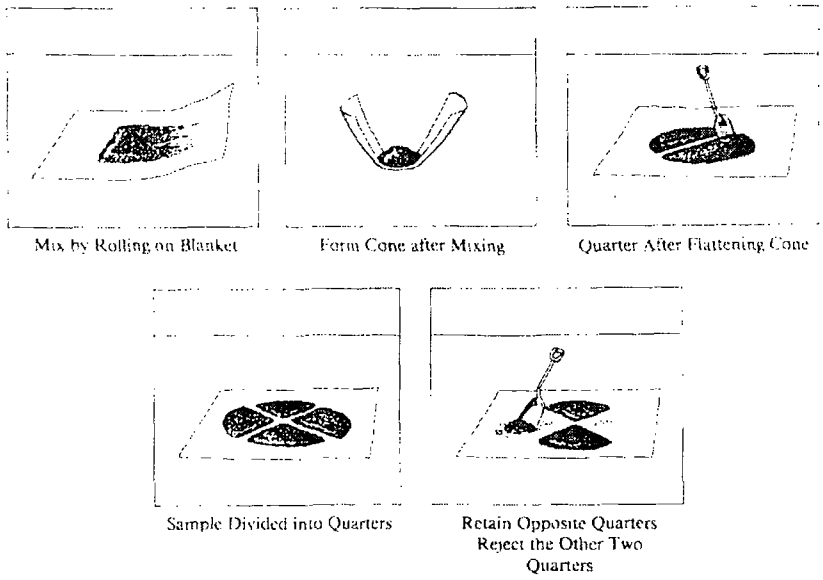


FIG. 4 Quartering on a Canvas Blanket

METHOD C—MINIATURE STOCKPILE SAMPLING (DAMP FINE AGGREGATE ONLY)

11. Apparatus

11.1 Apparatus shall consist of a straight-edged scoop, shovel, or trowel for mixing the aggregate, and either a small sampling thief, small scoop, or spoon for sampling.

12. Procedure

12.1 Place the original sample of damp fine aggregate on a hard clean, level surface where there will be neither loss of material nor the accidental addition of foreign material. Mix the material thoroughly by turning the entire sample over three times. With the last turning, shovel the entire sample into a conical pile by depositing each shovelful on top of the preceding one. If desired, flatten the conical pile to a uniform thickness and diameter by pressing down the apex with a shovel so that each quarter sector of the resulting pile will contain the material originally in it. Obtain a sample for each test by selecting at least five increments of material at random locations from the miniature stockpile, using any of the sampling devices described in 11.1.

13. Keywords

13.1 aggregate; aggregate—coarse; aggregate—fine; field testing—aggregate; sampling—aggregates; sample reduction; specimen preparation

INTERNATIONAL STANDARD

ISO 14966

First edition
2002-11-15

Ambient air — Determination of numerical concentration of inorganic fibrous particles — Scanning electron microscopy method

Air ambiant — Détermination de la concentration en nombre des particules inorganiques fibreuses — Méthode par microscopie électronique à balayage

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Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 14966 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 3, *Ambient atmospheres*.

Annexes A and B form a normative part of this International Standard. Annexes C, D and E are for information only.

Introduction

This International Standard describes a method for measurement of the numerical concentration of inorganic fibrous particles in ambient air using the scanning electron microscope. This International Standard is based on the procedures of Verein Deutscher Ingenieure (VDI) Guideline 3492 [6].

The method is also suitable for determining the numerical concentrations of inorganic fibres in the interior atmospheres of buildings, for example measurement of residual airborne fibre concentrations after the removal of asbestos-containing building materials [7].

Biological research has shown that the fibrogenic or carcinogenic effect of a fibre is related to its length, diameter and its resistance to dissolution in a biological environment. The point at which fibres are too short, too thick or of insufficient durability to produce a fibrogenic or carcinogenic effect is uncertain. Fibres with lengths greater than 10 µm and diameters of a few tenths of 1 µm, which also have durabilities such that they remain unchanged for many years in the body, are regarded as particularly carcinogenic. On the basis of current knowledge, fibres shorter than 5 µm are thought to have a low carcinogenic potential [8 to 11].

For the purposes of this International Standard, a fibre is defined as a particle which has a minimum length to width (aspect) ratio of 3:1. Fibres with lengths greater than 5 µm and widths extending from the lower limit of visibility up to 3 µm are counted. Fibres with diameters less than 3 µm are considered to be respirable. Since the method requires recording the lengths and widths of all fibres, the data can be re-evaluated if it is required to derive concentrations for fibres with a higher minimum aspect ratio [12].

The range of concentration to be measured extends from that found in clean air environments, in which the mean value of a large number of individual measurements of asbestos fibre concentrations has been found to be generally lower than 100 fibres/m^3 (fibres longer than 5 µm), up to higher exposure scenarios in which concentrations as much as two orders of magnitude higher have been found [10, 12].

This method is used to measure the numerical concentration of inorganic fibres with widths smaller than 3 µm and lengths exceeding 5 µm up to a maximum of 100 µm. Using energy-dispersive X-ray analysis (EDXA), fibres are classified as fibres with compositions consistent with those of asbestos fibres, calcium sulfate fibres and other inorganic fibres.

Calcium sulfate fibres are separated from other inorganic fibres and are not included in the final result, because on the basis of current knowledge, they do not represent any health hazard. Nevertheless, the numerical concentration of calcium sulfate fibres must be determined, since a high concentration of these fibres can negatively bias the results for probable asbestos fibres, and in some circumstances the sample may have to be rejected [13]. In addition, knowledge of the numerical concentration of calcium sulfate fibres is of importance in the interpretation of fibre concentrations in ambient atmospheres.

Detection and identification of fibres becomes progressively more uncertain as the fibre width is reduced below 0,2 µm. Identification of a fibre as a specific species is more confident if the source of emission is known or suspected, such as in a building for which bulk materials are available for analysis.

In order to facilitate the scanning electron microscope examination, organic particles collected on the filter are almost completely removed by a plasma ashing treatment.

Except in situations where fibre identification is difficult, there should be only minor differences between fibre counting results obtained by this method and those obtained using the procedures for determination of PCM-equivalent fibres in annex E of the transmission electron microscopy method ISO 10312:1995.

Ambient air — Determination of numerical concentration of inorganic fibrous particles — Scanning electron microscopy method

1 Scope

This International Standard specifies a method using scanning electron microscopy for determination of the concentration of inorganic fibrous particles in the air. The method specifies the use of gold-coated, capillary-pore, track-etched membrane filters, through which a known volume of air has been drawn. Using energy-dispersive X-ray analysis, the method can discriminate between fibres with compositions consistent with those of the asbestos varieties (e.g. serpentine and amphibole), gypsum, and other inorganic fibres. Annex C provides a summary of fibre types which can be measured.

This International Standard is applicable to the measurement of the concentrations of inorganic fibrous particles in ambient air. The method is also applicable for determining the numerical concentrations of inorganic fibrous particles in the interior atmospheres of buildings, for example to determine the concentration of airborne inorganic fibrous particles remaining after the removal of asbestos-containing products.

The range of concentrations for fibres with lengths greater than 5 µm, in the range of widths which can be detected under standard measurement conditions (see 6.2), is approximately 3 fibres to 200 fibres per square millimetre of filter area. The air concentrations, in fibres per cubic metre, represented by these values are a function of the volume of air sampled.

NOTE The ability of the method to detect and classify fibres with widths lower than 0,2 µm is limited. If airborne fibres in the atmosphere being sampled are predominantly < 0,2 µm in width, a transmission electron microscopy method such as ISO 10312 can be used to determine the smaller fibres.

2 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

2.1
acicular
shape shown by an extremely slender crystal with cross-sectional dimensions which are small relative to its length, i.e. needle-like

2.2
amphibole
any of a group of rock-forming double-chain silicate minerals, closely related in crystal form and composition, and having the nominal formula:

$$A_{0\text{-}1}B_2C_5T_8O_{22}(OH,F,Cl)_2$$

where

A = K, Na;

B = Fe^{2+}, Mn, Mg, Ca, Na;

C = Al, Cr, Ti, Fe^{3+}, Mg, Fe^{2+};

T = Si, Al, Cr, Fe^{3+}, Ti.

NOTE 1 See references [19] and [20].

NOTE 2 In some varieties of amphibole, these elements can be partially substituted by Li, Pb, or Zn. Amphibole is characterized by a cross-linked double chain of Si-O tetrahedra with a silicon:oxygen ratio of 4:11, by columnar or fibrous prismatic crystals and by good prismatic cleavage in two directions parallel to the crystal faces and intersecting at angles of about 56° and 124°.

2.3
amphibole asbestos
amphibole in an asbestiform habit

2.4
analytical sensitivity
calculated airborne fibre concentration equivalent to counting one fibre in the analysis

NOTE 1 It is expressed in fibres per cubic metre.

NOTE 2 This method does not specify a unique analytical sensitivity. The analytical sensitivity is determined by the needs of the measurement and the conditions found on the prepared sample.

2.5
asbestiform
specific type of mineral fibrosity in which the fibres and fibrils possess high tensile strength and flexibility

2.6
asbestos
any of a group of silicate minerals belonging to the serpentine and amphibole groups which have crystallized in the asbestiform habit, causing them to be easily separated into long, thin, flexible, strong fibres when crushed or processed

NOTE The Chemical Abstracts Service Registry Numbers of the most common asbestos varieties are: chrysotile (12001-29-5), crocidolite (12001-28-4), grunerite asbestos (amosite) (12172-73-5), anthophyllite asbestos (77536-67-5), tremolite asbestos (77536-68-6) and actinolite asbestos (77536-66-4).

2.7
asbestos structure
individual asbestos fibre, or any connected or overlapping grouping of asbestos fibres or bundles, with or without other particles

2.8
aspect ratio
ratio of length of a particle to its width

2.9
blank
fibre count made on a specimen prepared from an unused filter, to determine the background measurement

2.10
chrysotile
fibrous variety of the mineral serpentine, which has the nominal composition:

$Mg_3Si_2O_5(OH)_4$

NOTE Most natural chrysotile deviates little from this nominal composition. In some varieties of chrysotile, minor substitution of silicon by Al^{3+} may occur. Minor substitution of magnesium by Al^{3+}, Fe^{2+}, Fe^{3+}, Ni^{2+}, Mn^{2+} and Co^{2+} may also be present. Chrysotile is the most prevalent type of asbestos.

2.11
cleavage
breaking of a mineral along one of its crystallographic directions

2.12
cleavage fragment
fragment of a crystal that is bounded by cleavage faces

2.13
cluster
fibrous structure in which two or more fibres, or fibre bundles, are randomly oriented in a connected grouping

2.14
countable fibre
any object longer than 5 µm, having a maximum width less than 3 µm and a minimum aspect ratio of 3:1

2.15
energy-dispersive X-ray analysis
measurement of the energies and intensities of X-rays by use of a solid-state detector and multi-channel analyser system

2.16
field blank
filter cassette which has been taken to the sampling site, opened and then closed, and subsequently used to determine the background fibre count for the measurement

2.17
fibre
elongated particle which has parallel or stepped sides and a minimum aspect ratio of 3:1

2.18
fibre bundle
structure composed of apparently attached, parallel fibres

NOTE A fibre bundle may exhibit diverging fibres at one or both ends. The length is defined as equal to the maximum length of the structure, and the diameter is defined as equal to the maximum width in the compact region.

2.19
fibril
single fibre of asbestos which cannot be further separated longitudinally into smaller components without losing its fibrous properties or appearances

2.20
fibrous structure
fibre, or connected grouping of fibres, with or without other particles

2.21
habit
the characteristic crystal growth form or combination of these forms of a mineral, including characteristic irregularities

2.22
image field
the area on the filter sample which is shown on the cathode ray tube display

2.23
limit of detection
calculated airborne fibre concentration equivalent to the upper 95 % confidence limit of 2,99 fibres predicted by the Poisson distribution for a count of zero fibres

NOTE It is expressed in fibres per cubic metre.

2.24
magnification
ratio of the size of the image of an object on the cathode ray tube screen to the actual size of the object

NOTE For the purposes of this International Standard, magnification values always refer to that applicable to the cathode ray tube display.

2.25
matrix
structure in which one or more fibres or fibre bundles touch, are attached to, or partially concealed by a single particle or connected group of non-fibrous particle

2.26
serpentine
any of a group of common rock-forming minerals having the nominal formula:

$Mg_3Si_2O_5(OH)_4$

2.27
split fibre
agglomeration of fibres which, at one or several points along its length, appears to be compact and undivided, whilst at other points appears to separate into separate fibres

2.28
structure
single fibre, fibre bundle, cluster or matrix

3 Abbreviated terms

CRT Cathode ray tube

EDXA Energy-dispersive X-ray analysis

FWHM Full width, half maximum

PTFE Polytetrafluoroethylene

SEM Scanning electron microscope

UICC Union Internationale Contre le Cancer

4 Principle

A sample of airborne particulate is collected by drawing a measured volume of air through a gold-coated, capillary-pore track-etched membrane filter with a maximum nominal pore size of 0,8 µm, which is subsequently examined in the scanning electron microscope (SEM). Before analysis, the gold-coated filter is treated in a plasma asher to remove organic particles, to the extent that this is possible. The individual fibrous particles and constituent fibres in a randomly-selected area of the filter are then counted at a magnification of approximately 2 000 ×. If a fibre is detected at the magnification of approximately 2 000 ×, it is examined at a higher magnification of approximately 10 000 × to measure its dimensions. At the higher magnification of approximately 10 000 ×, energy-dispersive X-ray analysis (EDXA) is used to classify the fibre according to the chemical composition.

The limit of detection for this method is defined as the numerical fibre concentration below which, with 95 % confidence, the actual concentration lies when no fibres are found during the SEM examination. The limit of detection theoretically can be lowered indefinitely by filtration of progressively larger volumes of air and by examination of a larger area of the specimen in the SEM. In practice, the lowest achievable limit of detection for a particular area of SEM specimen examined is controlled by the total suspended particulate concentration remaining after the plasma ashing step.

A limit of detection of approximately 300 fibres/m^3 is obtained if an air volume of 1 m^3 per square centimetre of filter surface area passes through the filter, and an area of 1 mm^2 of the filter area is examined in the SEM. This corresponds to an evaluated sample air volume of 0,01 m^3.

5 Apparatus and materials

5.1 Air sampling

5.1.1 Sampling head.

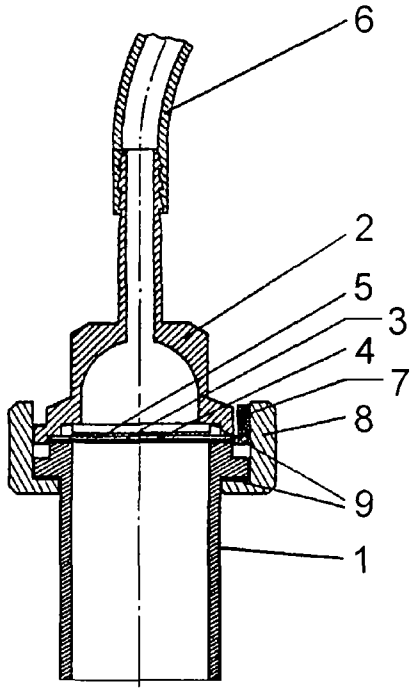
A disposable, 3-piece, conductive plastic field monitor cassette may be used as the sampling head, provided that the design is such that significant leakage around the filter does not occur. A re-usable unit may also be used as the sampling head, consisting of a cylindrical cowl and a filter holder with backing filter. Figure 1 shows an example of a suitable sampling head. The cowl and the filter holder should be made from a corrosion-resistant material. The filter must be clamped in such a manner that significant leaks around the filter do not occur at differential pressures up to approximately 50 kPa (see B.4). The length of the cowl should be 0,5 to 2,5 times the effective filter diameter (the diameter of the exposed circular filter area through which the air is drawn). If it is possible that wind velocities greater than 5 m/s could occur during sampling, use a long cowl with a ratio of length to effective diameter of 2,5.

5.1.2 Sampling train.

Figure 2 shows an example of a suitable sampling train. Control of the volumetric flowrate may be achieved either by the use of a throttle valve (3) or a volumetric flow controller (8) in conjunction with a regulator valve (4).

5.1.3 Sampling pump, pulse-free or pulsation-damped, capable of maintaining, at a pressure differential across the filter of at least 50 kPa, a volumetric flow of between 8 l/min and 30 l/min, depending on the diameter of filter used.

In order to achieve the required analytical sensitivity, a flowrate of 8 l/min is required if a 25 mm diameter filter is used. This flowrate is equivalent to a filter face velocity of approximately 35 cm/s, which results in a pressure differential of approximately 50 kPa. The sampling pump shall be capable of maintaining the intended flowrate within ± 10 % throughout the whole sampling period.



Key

1	Cowl	6	Suction hose
2	Filter holder	7	Clamping roller
3	Backing filter	8	Clamping ring
4	Track-etched membrane filter	9	PTFE gaskets
5	Supporting mesh		

Figure 1 — Example of design of sampling head

5.1.4 **Needle valve**, with a fine adjustment mechanism, for setting the volumetric flowrate.

5.1.5 **Volumetric flowmeter** (rotameter), for measuring the volumetric flowrate.

5.1.6 **Timer**, for measuring the sampling time.

5.1.7 **Dry type gas meter** (optional), for volumetric measurement, calibrated, designed for a maximum volumetric flowrate of 2 m^3/h.

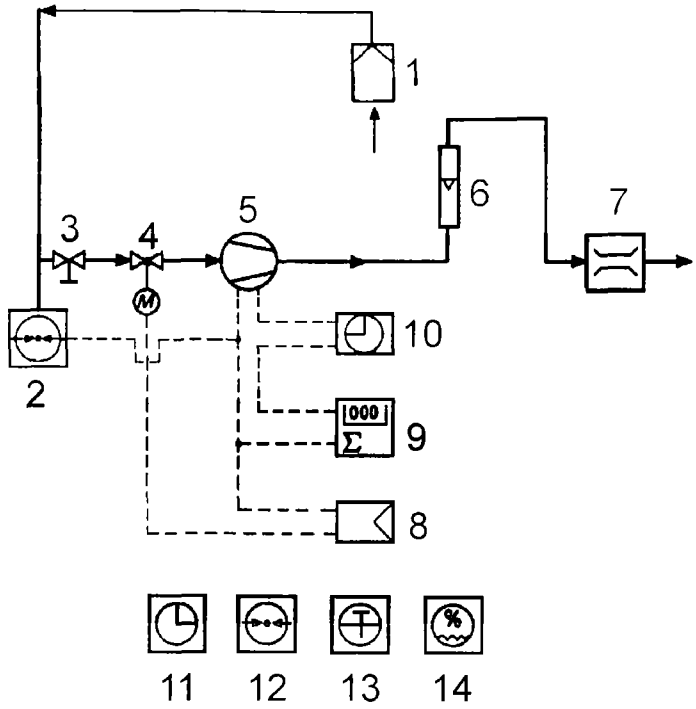
5.1.8 **Meteorological instruments** (optional), for recording of meteorological conditions during sampling.

Instruments such as a thermometer, a hygrometer, a barometer and a wind speed and direction recorder will be required.

5.1.9 **Instruments for unattended sampling** (optional).

For unattended sampling, a volumetric flow controller is required for regulation of the flowrate to within ± 10 % of the nominal rate, with an automatic switch to turn off the sampling pump if the flowrate exceeds or falls below the pre-set tolerance band. The flow controller can be integrated into the suction unit.

A programmable switch is required for pre-setting the air sampling cycle. A pressure gauge which incorporates a switching contact is required to switch off the sampling pump if the pressure differential across the sampling filter increases to a pre-set value.



Key

1 Sampling head or cassette
2 Pressure gauge
3 Throttle valve (optional)
4 Regulator valve (optional)
5 Pump
6 Variable-area flowmeter
7 Gas meter (optional) with thermometer
8 Volumetric flow controller (optional)
9 Sampling-time recorder (optional)
10 Programmer (optional)
11 Timer
12 Thermometer (optional)
13 Barometer (optional)
14 Hygrometer (optional)

Figure 2 — Example of a suitable sampling train

5.2 Preparation of filters

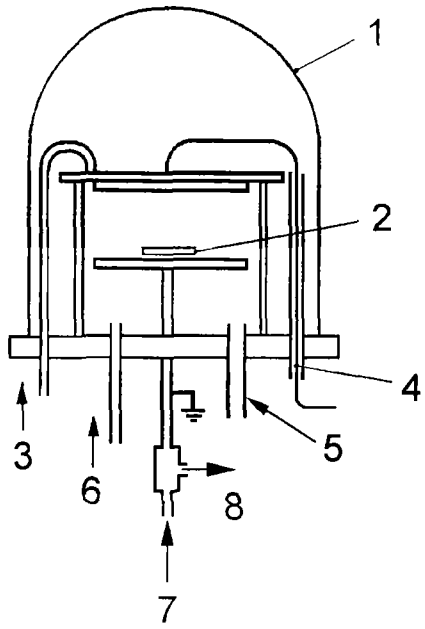
5.2.1 Vacuum evaporator, capable of producing a vacuum better than 0,013 Pa.

A vacuum coating unit is required for vacuum deposition of gold onto the capillary-pore membrane filters, and for carbon coating of SEM specimens if the particulate loading is such that excessive charging of the specimen occurs.

A sputter coating unit has also been found to meet the requirements for gold coating of the capillary-pore filters.

5.2.2 Plasma asher, supplied with oxygen, to oxidize organic particles on the SEM specimen.

An example of the configuration of a suitable plasma asher is shown in Figure 3. The chamber of the plasma asher may be coupled either capacitatively or inductively. Care shall be taken not to damage the specimen during the plasma ashing process. A calibration procedure to determine suitable operating conditions for the plasma asher is described in B.3.



Key

1	Bell jar	5	Connection for vacuum pump
2	Filter in mounting ring	6	Air inlet
3	Oxygen inlet	7	Cooling-water inlet
4	Power supply from plasma generator	8	Cooling-water outlet

Figure 3 — Example of a configuration of a plasma asher

5.3 Sample analysis

5.3.1 Scanning electron microscope (SEM), with an accelerating voltage of at least 20 kV, is required for fibre counting and identification.

5.3.2 Energy-dispersive X-ray system for the SEM, capable of achieving a resolution better than 170 eV (FWHM) on the MnK_a peak.

The performance of an individual combination of SEM and solid-state X-ray detector is dependent on a number of geometrical factors. Accordingly, the required performance of the combination of the SEM and X-ray analyser is specified in terms of the measured X-ray intensity obtained from a chrysotile fibre of width 0,2 µm, under the operating conditions used during the analysis. Solid-state X-ray detectors are least sensitive in the low energy region, and so detection of sodium in crocidolite is an additional performance criterion.

The instrumental combination shall satisfy the minimum requirements with regard to the visibility of fibres, as specified in 6.4.1, and identification of the fibres, as specified in 6.4.3.

5.3.3 Stereo-microscope, with a magnification of approximately 20 ×, for visual examination of the particulate deposit on the filter.

5.3.4 Gold-coated capillary-pore polycarbonate filters, of 0,8 µm maximum nominal pore size, for collection of air samples.

The gold coating shall be approximately 30 nm thick applied to the shiny side of the filter. The procedure for preparation of the gold-coated filters is described in annex A.

NOTE Optionally, a 20 nm thick layer of gold may be evaporated on to the reverse side of the filter. This coating serves to protect the filter during plasma ashing and can help to improve the contrast of fibres in the SEM image.

5.3.5 Backing filters of cellulose ester membrane, or absorbent pads, with a porosity of approximately 5 µm to be used as diffusing filters behind the sample collection filters.

5.3.6 Disposable plastic field monitors (optional).

If disposable plastic field monitors are used, they shall consist of 25 mm to 50 mm diameter, three-piece cassettes, which conform to the requirements of 5.1.1. The cassette shall be loaded with a gold-coated, capillary-pore polycarbonate filter of maximum nominal pore size 0,8 µm, backed by a cellulose ester filter of 5 µm porosity. Suitable precautions shall be taken to ensure that the filters are tightly clamped in the assembly so that significant air leakage around the filter cannot occur.

Re-use of disposable plastic field monitors is not recommended.

5.3.7 Technically pure oxygen, for operation of the plasma asher.

5.3.8 Rubber connecting hoses, for connecting the sampling head to the pump, and other equipment in the sampling train.

The hose shall have a wall thickness such that it does not collapse under a vacuum of 50 kPa. Silicone rubber hose has been found to meet the requirements.

5.3.9 Filter containers, for transport and storage of filters if disposable field monitors are not used.

5.3.10 Routine electron microscopy tools and supplies.

Fine point tweezers, scalpel holders and blades, double-coated adhesive tape, SEM specimen stubs and colloidal carbon paint and other routine supplies are required. If a vacuum evaporator is used for preparation of gold-coated filters, gold wire and tungsten filaments are required. For carbon evaporation, spectroscopically pure carbon rods and a means of sharpening the rods is required.

5.3.11 Sample for resolution adjustment.

A gold-coated polycarbonate filter, on which chrysotile fibres with a width $< 0{,}2$ µm have been deposited, is required for adjustment of the operating conditions of the SEM.

5.3.12 Sample for magnification calibration

A test sample is required to calibrate the magnification of the SEM. The magnification standard SRM484e (U.S. National Institute of Standards and Technology) is an example of a sample which meets the requirement.

6 Air sample collection and analysis

6.1 Measurement planning

When determining the spatial and temporal scope of the measurements, it is important to take into consideration the special aspects of the situation. It is therefore essential to define the objective of the measurements before samples are collected. Any available information on emission sources, meteorological conditions and the local situation should be taken into account in order to obtain the maximum information from the measurements. The number of individual measurements to be made should be selected according to the particular task. In particular, prior to collection of the samples, the required accuracy for the mean concentration of the inorganic fibres should be specified, since the error of each individual measurement needs to be taken into consideration in determining the number of samples to be collected. Measurement uncertainty is discussed in clause 8.

6.2 Collection of air samples

Figure 2 shows an example of a sampling train. Position the sampling head approximately 1,5 m above ground level.

If a re-usable sampling head is used, place a 5 µm porosity cellulose ester backing filter on to the filter support in the sampling head. Place a gold-coated filter on top of the backing filter, with the shiny side facing into the direction of the airflow. Clamp the filters in the sampling head so that the gold-coated filter lies flush against the backing filter and is tightly fitted. Ensure that damage does not occur to the filter during clamping, and that the filter is not twisted.

Before air sampling is commenced, perform a brief test with the tube to the sampling head closed, to determine if any leaks exist in the complete sampling system. Under the conditions of this test, the flowrate indicated by the volumetric flowmeter shall be less than 10 % of the unimpeded flowrate. Open the tube only after the pump has been switched off, in order to avoid sudden pressure surges.

Leaks around the filter can also occur if the filter is inadequately sealed on the low pressure side, or if the filter has been damaged. Observation of a lower differential pressure at the start of the air sampling indicates that a serious leak exists. If, after sampling, particulate deposits are observed around the edge of the backing filter or on the unexposed edges of the sampling filter, a leak around the filter has occurred and the sample shall be rejected.

When sampling is to be commenced, start the pump and the timer simultaneously.

Within 2 min of the start of sampling, adjust the volume flowrate to approximately 2 l/min per square centimetre of effective filter area (this value shall not vary by more than ± 10 % for the period of sampling). This corresponds to a filter loading of 1 000 l per square centimetre of effective filter area over a sampling period of approximately 8 h.

At the end of the sampling period, switch off the sampling apparatus. If a programmer was used, confirm that the sampler operated within the required parameters for the preset sampling period. Taking care not to disturb the particulate deposits on the filter surface, remove the sample collection filter and store it upright in a dust-tight sample container.

Record all sampling data which may be of significance for later interpretation. An example of a form for recording of air sampling data is shown in Figure 4. The location of the sampling apparatus shall be documented in the form of a sketch and, if possible, a photograph.

In fog, a thick coating (including calcium sulfate fibres) on the sampling filter may occur, resulting in a rapid increase in the pressure differential across the filter. Under these conditions, it is not possible to collect a satisfactory sample to represent the normal sampling period, and it will be necessary to take several sequential samples, each collected over a shorter sampling time, in order to obtain filters suitable for analysis. Annex E shows the procedure for calculation of a mean value from the results of several sequential short-term samples. If fog is an unusual occurrence, more representative results could be obtained by collection of air samples when the weather conditions are more typical.

6.3 SEM specimen preparation

Before sample analysis, examine the uniformity of the particulate deposit on the filter. If the particulate deposit shows evidence of non-uniformity, reject the filter.

If the particulate deposit is uniform, place the filter into the holder of the mounting ring, and position it in the plasma asher, as shown in Figure 3. The plasma ashing treatment removes the majority of the organic material on the filter, and this considerably facilitates the SEM analysis of the sample.

The rate of oxidation of the organic material on the filter by the oxygen plasma is enhanced by the electrical conductivity of the filter and the sample holder. Under the specified operating conditions, the plasma ashing treatment is generally completed after approximately 30 min. After the plasma ashing treatment, either the whole filter or a part thereof is mounted on an SEM specimen stub, without any further preparation, for SEM analysis.

NOTE 1 The portion of the filter to be analysed may be mounted on the SEM specimen stub either before or after the plasma ashing treatment.

NOTE 2 Double-sided conductive adhesive tape has been found to be an effective means of mounting the filter.

AIR SAMPLING DATA SHEET: ISO 14966	
Project:	**No.:**
Sample No.:	
Sampling location:	
Sampling apparatus (type):	
Sampling times:	
Start (date, time):	End (date, time):
Duration (hours, minutes):	
Sampling filter (type):	
Nominal pore size (µm):	Diameter (mm):
Effective diameter (mm):	Effective filter area (mm^2):
Sampling data	
Volumetric meter readings:	
Start (m^3):	End (m^3):
Volumetric throughput (m^3):	
Volumetric flowrate	
Start (m^3/h):	End (m^3/h):
Mean volumetric flowrate (m^3/h):	
Meteorological data (if required)	
Air temperature (°C):	Relative humidity (%):
Wind velocity (m/s):	
Weather characteristics:	
Remarks:	
Sampler (Name): **Signature:** _____	**Date of analysis:**

Figure 4 — Example of a sampling log form for recording of sampling data

If, during SEM analysis, fibres are detected which appear to be organic, the plasma ashing treatment can be repeated to remove them.

In exceptional cases, it may be necessary to evaporate a thin film of carbon onto the SEM specimens in order to reduce localized charging, increase the contrast, and thus improve the visibility of fine fibres. This will generally be required only when the filter has a very heavy particulate loading.

6.4 Analysis in the scanning electron microscope

6.4.1 General instructions

Examine the filter sample at an accelerating voltage of approximately 20 kV and an image magnification of between 2 000 × and 2 500 ×. For fibre classification in the SEM, an accelerating voltage of 20 kV is recommended.

Adjust the SEM such that fibres with a width of approximately 0,2 µm are just visible at a magnification of 2 000 ×. This adjustment is performed by selecting a fibre on the prepared sample, or on a test sample, which is just visible at the magnification of approximately 2 000 × used for fibre counting. The width of this fibre is then confirmed by measuring it at a magnification of 20 000 ×. This adjustment shall be carried out on at least two separate fibres before the analysis is started, and it shall be repeated several times during the course of a series of analyses to ensure that the fibre visibility conditions have not changed.

Position the X-ray detector such that it subtends the largest possible solid angle at the specimen surface. The sample shall not be tilted to an angle greater than 20° when counting and sizing the fibres.

Select the operating parameters of the SEM and the X-ray detector system so that a statistically acceptable X-ray spectrum can be acquired from a chrysotile fibre of 0,2 µm width on the test sample within a maximum period of 100 s.

The criteria for statistical acceptability require, for peak height, P, and background level, B:

$$P > 3\sqrt{B} \tag{1}$$

with a minimum of 30 pulses in the channel corresponding to the maximum peak height for each of the magnesium and silicon peaks [16], and

$$\frac{P+B}{B} > 2 \tag{2}$$

for each of the magnesium and silicon peaks.

During analysis, each selected image field is examined for fibres of the length and width ranges specified in 6.4.2. Using EDXA, these fibres are then classified into compositional groups according to the criteria specified in 6.4.3. The sequential number of the image field, the fibre length, the fibre width, the elemental composition and the fibre classification are recorded on a fibre counting form. An example of a suitable fibre counting form is shown in Figure 5. In order to document the appearance and particulate loading of the sample, three micrographs shall be taken of each sample and attached to the fibre-counting form.

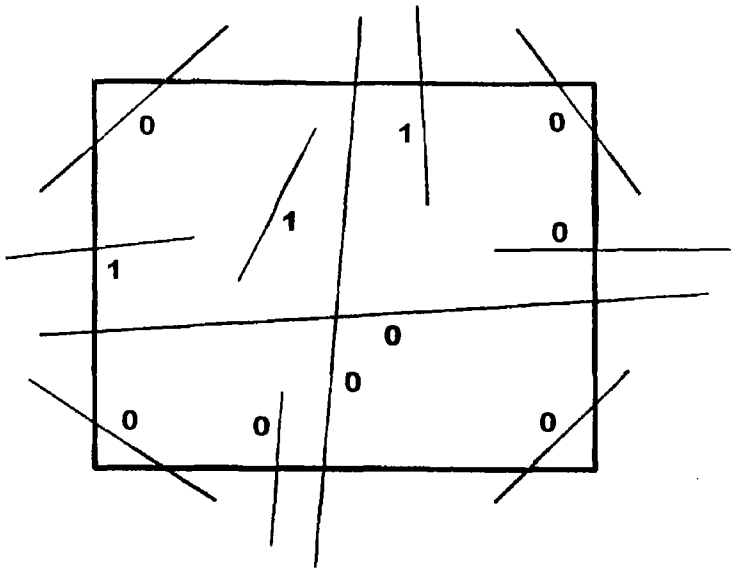
6.4.2 Fibre-counting criteria

6.4.2.1 General

Examine at least 50 image fields in order to reduce, to the extent that is possible, the effect of fluctuations in the filter loading density on the counting result. Select image fields to be evaluated in such a way that the whole area of the sample is taken into account and overlapping of the image fields does not occur. Count fibres in accordance with the specifications in 6.4.2.2 to 6.4.2.9, and the examples shown in Figures 6 and 7.

FIBRE COUNTING FORM: ISO 14966						
Sample No.:			**Date:**		**Page No.:**	**Name:**
Calcium sulfate	**Tally list:**				**Rejected structures**	**Tally list:**
Number of image fields	**Fibre No.**	**Image field No.**	*l* µm	*D* µm	**Elemental composition**	**Fibre type**
Tally list:	1					
	2					
	3					
	4					
	5					
	6					
	7					
	8					
	9					
	10					
	11					
	12					
	13					
	14					
	15					
	16					
	17					
	18					
	19					
	20					
	21					
	22					
	23					
	24					
	25					
	26					
	27					
	28					
	29					
	30					
Totals	Chrysotile type fibres: Amphibole fibres: Other inorganic fibres: Calcium sulfate:				Number rejected: Bundles: Clusters: Matrices: Number of fibres without spectrum:	
Total number of image fields: Number of image fields rejected: Calibrated magnification:					Micrograph numbers: 1: 2: 3:	

Figure 5 — Example of fibre counting form

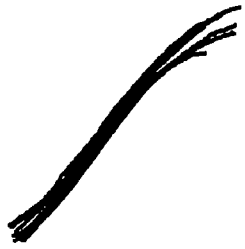


0 = Do not count
1 = Count

Figure 6 — Examples of fibres extending outside the image field

Fibre: count as 1

Split fibre: count as 1

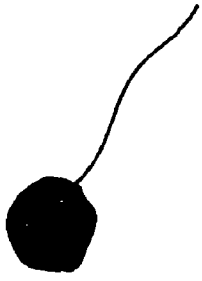


Fibre bundle: count as 1

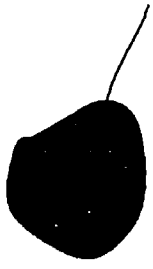
Cluster: count as 3

Cluster: count as 5

Matrix: count as 1 (particle < 3 µm)



Matrix: count as 1 (particle < 3 µm)



Do not count (particle > 3 µm)

Matrix: count as 2 (particle < 3 µm)

Figure 7 — Examples of the interpretation of the counting rules

6.4.2.2 Countable fibre

A countable fibre is defined in 2.14 as any object longer than 5 µm, a maximum width less than 3 µm, and a minimum aspect ratio of 3:1.

6.4.2.3 Bundle

A fibre bundle or a split fibre is counted as a single fibre if the overall dimensions of the bundle or the split fibre conform to those of the countable fibre definition as given in 2.14. The diameter of a fibre bundle or split fibre is defined as the maximum width in the compact region.

6.4.2.4 Cluster

Each countable fibre, fibre bundle or split fibre within a cluster is counted individually if both ends of the fibre, bundle or split fibre can be separately located and its length and maximum width measured. For a cluster in which no individual countable fibres are visible, count the cluster as a single fibre if the overall dimensions of the cluster conform to those of the countable fibre definition in 2.14.

6.4.2.5 Matrix

Each countable fibre, fibre bundle or split fibre within a matrix is counted individually if both ends of the fibre, bundle or split fibre can be separately located and its length and maximum width measured. For a matrix in which no individual countable fibres are visible, count the matrix as a single fibre if overall dimensions of the matrix conform to those of the fibre definition in 2.14.

6.4.2.6 Fibres extending outside of the image field

For each image field, count all countable fibres except those which extend over the right-hand or the bottom edge of the image field. Do not count fibres which have no terminations within the image field. Figure 6 shows examples of the counting criteria.

6.4.2.7 Rejection of overloaded image fields

Reject the image field if more than one eighth of an image field is covered by aggregates of fibres and/or particles. Record each rejection of an image field on the fibre counting form. If more than 10 % of the image fields are rejected in accordance with this criterion, the sample is overloaded and shall be rejected.

6.4.2.8 Recording of fibrous particles not meeting the specified definitions

Observations of fibrous particles which do not meet the specified definitions, for example, fibres with adherent particles (matrices), or fibre bundles and clusters with widths greater than 3 µm, shall be recorded on the fibre counting form.

6.4.2.9 Termination of fibre counting

Continue the examination until completion of the field in which the 100th inorganic fibre (other than calcium sulfate fibres) occurs. If, after examination of 50 image fields, 100 fibres have not been detected, further fields shall be examined until either a total of 100 inorganic fibres has been counted or sufficient area has been examined to achieve the desired analytical sensitivity. For most applications, it is recommended that at least 1 mm^2 of the filter area be examined.

6.4.3 Fibre classification

6.4.3.1 General

A simple fibre classification procedure is used in which each fibre is classified as chrysotile asbestos, amphibole asbestos, calcium sulfate or other type of inorganic fibre on the basis of a semi-quantitative analysis of the elemental spectrum. The fibres are classified into the above categories on the basis of their X-ray emission spectra.

NOTE In this method, the term "fibre classification" is used, rather than "fibre identification", to distinguish between a definitive identification based on a combination of morphology, chemical composition and crystal structure, and the result obtained by this method in which fibres are presumed to be asbestos if they exhibit an EDXA spectrum consistent with one of the asbestos varieties. If airborne fibres detected by this method exhibit EDXA spectra consistent with bulk materials present at the site where the air samples were collected, and these bulk materials have already been identified as one of the asbestos varieties by polarized light microscopy or transmission electron microscopy, the presumption that the fibres observed are asbestos is much stronger.

Fibres are classified into four categories:

a) fibres with chemical compositions consistent with those of serpentine asbestos;

b) fibres with chemical compositions consistent with those of amphibole asbestos;

c) calcium sulfate fibres;

d) other inorganic fibres.

The "other inorganic fibres" category includes all fibres which cannot be classified as either asbestos or calcium sulfate, but which do exhibit spectra indicating that they are of inorganic compositions.

It is important to recognize that, during acquisition of an EDXA spectrum from a fibre, scattering of the electron beam may result in emission of X-rays from particles attached to, or in close proximity to the fibre being analysed. The EDXA spectrum obtained may therefore contain contributions from these particles, and the spectrum may contain X-ray peaks from elements that are not present in the asbestos varieties. In these cases, attempts should be made to acquire EDXA spectra from several positions on the fibre, as far away from adhering or adjacent particles or fibres as possible, in order to minimize the contributions from the other particles.

Use the criteria given in 6.4.3.2 to 6.4.3.8 to classify the spectra:

6.4.3.2 Serpentine (chrysotile)

Classify a fibre as serpentine (chrysotile) if:

a) the Mg and Si peaks are clear, with $(P + B)/B > 2$;

b) any Fe, Mn and Al peaks are small, with $P/B < 1$.

NOTE 1 Depending on the composition of adjacent or attached particles, other peaks such as Ca or Cl may also be visible.

NOTE 2 Anthophyllite and talc both yield EDXA spectra which conform to this specification, but the Mg/Si peak height ratio for these minerals is lower than that for serpentine. In order to avoid erroneous classification of talc or anthophyllite as serpentine, it is important to take account of the Mg/Si peak height ratio, and to calibrate the EDXA detector using known samples of serpentine and talc.

6.4.3.3 Amosite

Classify a fibre as amosite if:

a) the Si and Fe peaks are clear, with $(P + B)/B > 2$;

b) any Na, Mg and/or Mn peaks are small.

NOTE Depending on any adjacent or attached particles, other peaks such as Ca or Cl may also be visible.

6.4.3.4 Crocidolite

Classify a fibre as crocidolite if:

a) the Na, Si and Fe peaks are clear, with $(P + B)/B > 2$;

b) any peak from Mg is small, and any Mn peak is small with $P/B < 1$.

NOTE Depending on any adjacent or attached particles, other peaks such as Ca or Cl may also be visible.

6.4.3.5 Tremolite or actinolite

Classify a fibre as tremolite or actinolite if:

a) the Mg, Si and Ca peaks are clear, with $(P + B)/B > 2$;

b) a peak from Fe may be present, but any Na peak is faint, with $P/B < 1{:}1$.

NOTE Depending on any adjacent or attached particles, other peaks such as Ca or Cl may also be visible.

6.4.3.6 Anthophyllite or talc

Classify a fibre as anthophyllite or talc if:

a) the Mg and Si peaks are clear, with $(P + B)/B > 2$;

b) the Mg/Si peak height (or area) ratio is consistent with that obtained on fibres of reference anthophyllite or talc, and any peaks from Fe, and Ca are small.

NOTE Using this analytical method, it is not possible to discriminate routinely between anthophyllite with a low iron concentration and talc with a high iron concentration. The fibre morphology may assist in discrimination between anthophyllite and talc. Ribbon-like fibres are probably talc, whereas straight, rod-like fibres are possibly, but not necessarily, anthophyllite. If fibres of this composition are observed, it is recommended that the sample be evaluated using transmission electron microscopy.

6.4.3.7 Calcium sulfate

Classify a fibre as calcium sulfate if:

— the Ca and S peaks are clear, with $(P + B)/B > 2$.

NOTE Depending on any adjacent or attached particles, peaks from other elements may be visible.

6.4.3.8 Other inorganic fibres

Classify a fibre as an other inorganic fibre if it yields a spectrum containing any combination of elements which cannot be classified into categories 6.4.3.2 to 6.4.3.7.

NOTE 1 On the basis of the above criteria, silicate fibres can be classified only as those exhibiting chrysotile-like or amphibole-like elemental spectra (asbestos), calcium sulfate and other inorganic fibres. This procedure can result in an over-estimation of the asbestos fibre content [12].

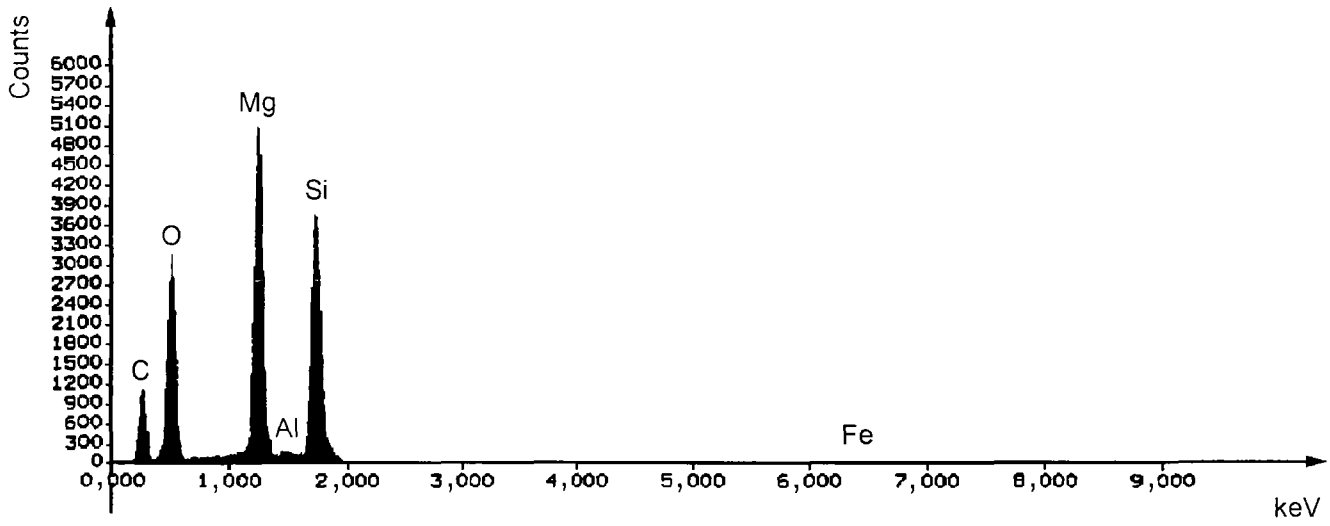
NOTE 2 In the above criteria, amosite and crocidolite have been classified separately. However, some X-ray detectors may not be sufficiently sensitive to detect the sodium peak from crocidolite and, when using these types of detector, routine discrimination between amosite and crocidolite may not be possible. In this case, amosite and crocidolite fibres are classified into the same category.

6.4.3.9 Fibres which exhibit no definitive X-ray peaks

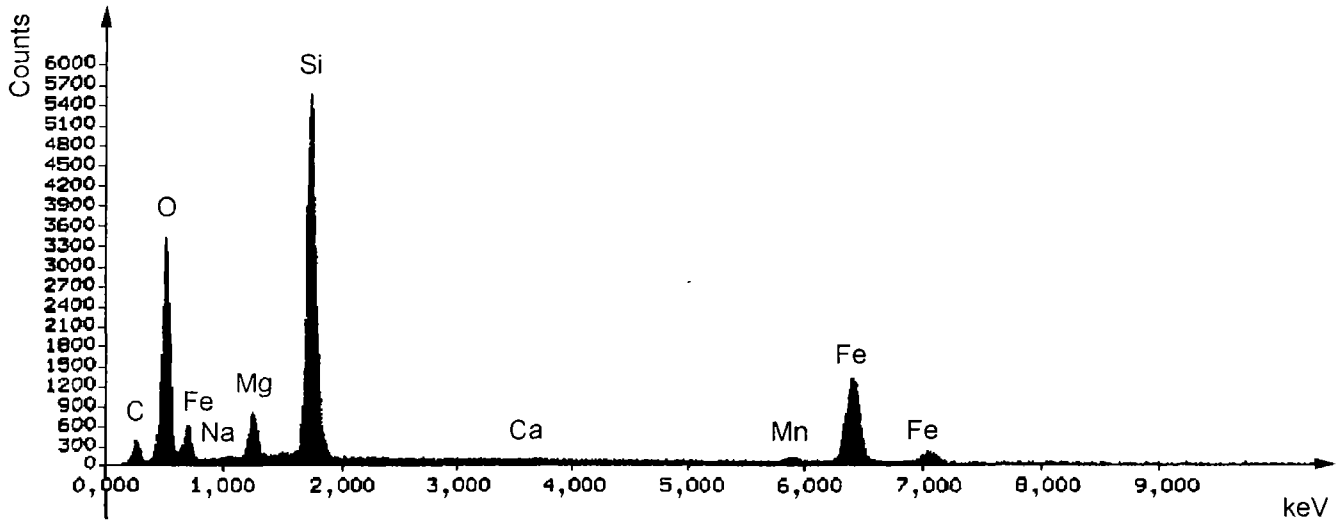
Observation of fibres which yield no definitive X-ray peaks in the EDXA spectrum can be interpreted as an indication that residual organic material is still present after ashing. Very fine inorganic fibres with widths less than 0,2 µm usually do not yield statistically significant X-ray peaks.

6.4.3.10 Reference EDXA spectra from standards of the asbestos varieties

For any particular fibre, the relative heights of the peaks in the EDXA spectrum vary with the characteristics of the X-ray detector. In particular, the detection efficiency for X-ray peaks from low atomic number elements is higher for ultra-thin window detectors than it is for standard beryllium window detectors. Because each EDXA detector has different efficiency characteristics, it is necessary to obtain reference spectra for each SEM-EDXA system, using standards of the asbestos varieties. A series of such spectra, collected using an ultra-thin window detector, are shown as examples in Figure 8. These spectra are used for comparison purposes in the classification of fibres. Since the performance of the EDXA detector may change with time, new reference spectra must be obtained at appropriate intervals, and particularly after any maintenance of the detector has been carried out.

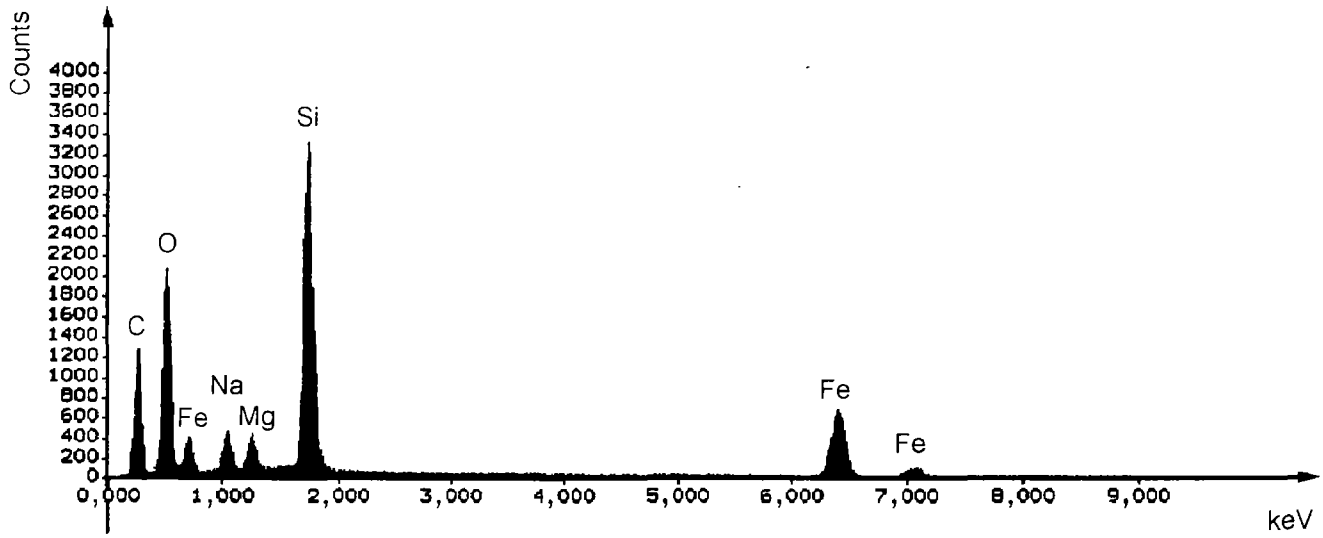


a) Fibre of reference chrysotile (no gold coating)

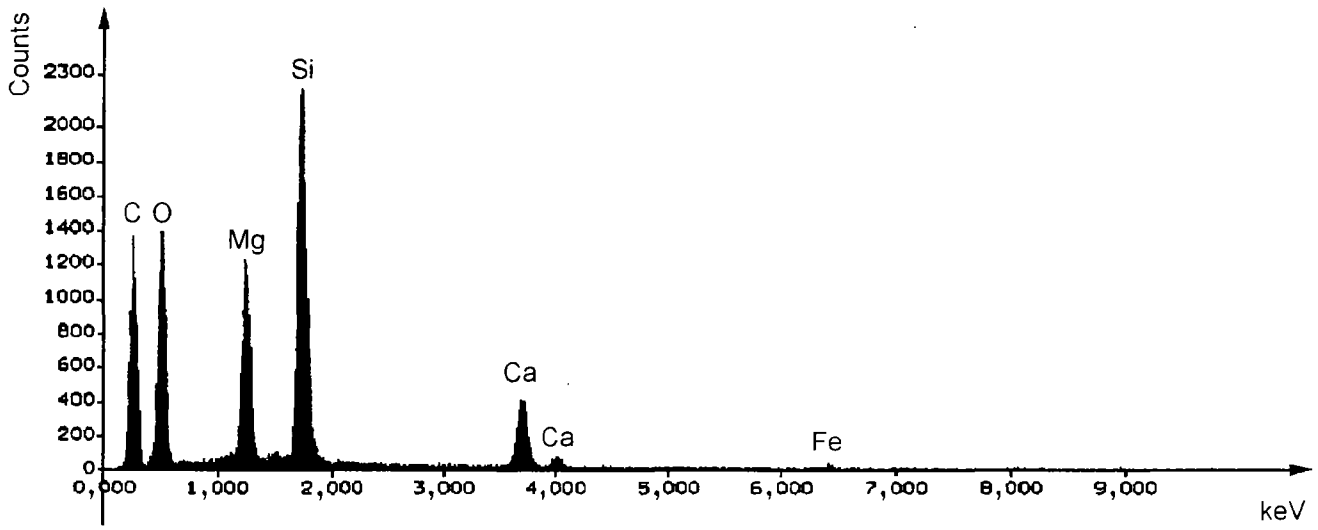


b) Fibre of reference amosite (no gold coating)

Figure 8 — EDXA spectra of fibres of reference materials

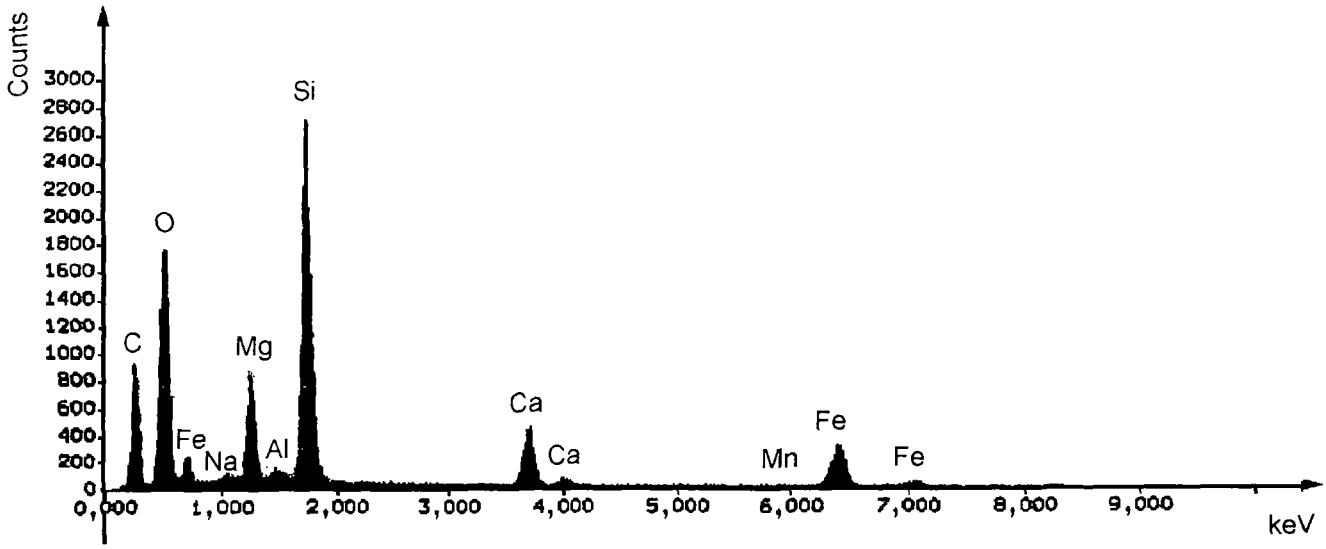


c) **Fibre of reference crocidolite (no gold coating)**

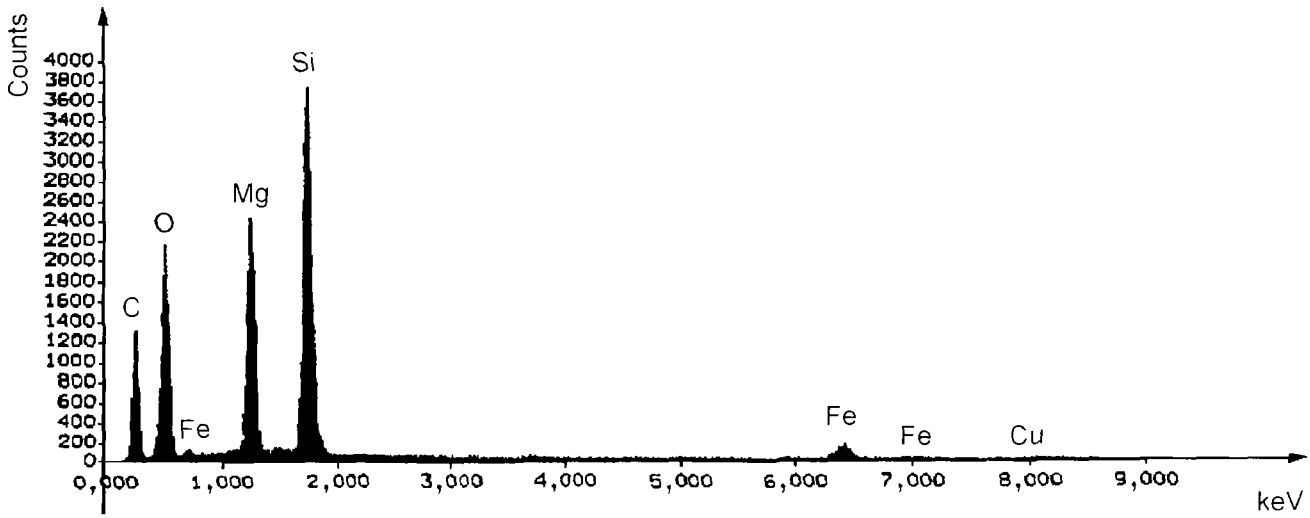


d) **Fibre of reference tremolite (no gold coating)**

Figure 8 (*continued*)

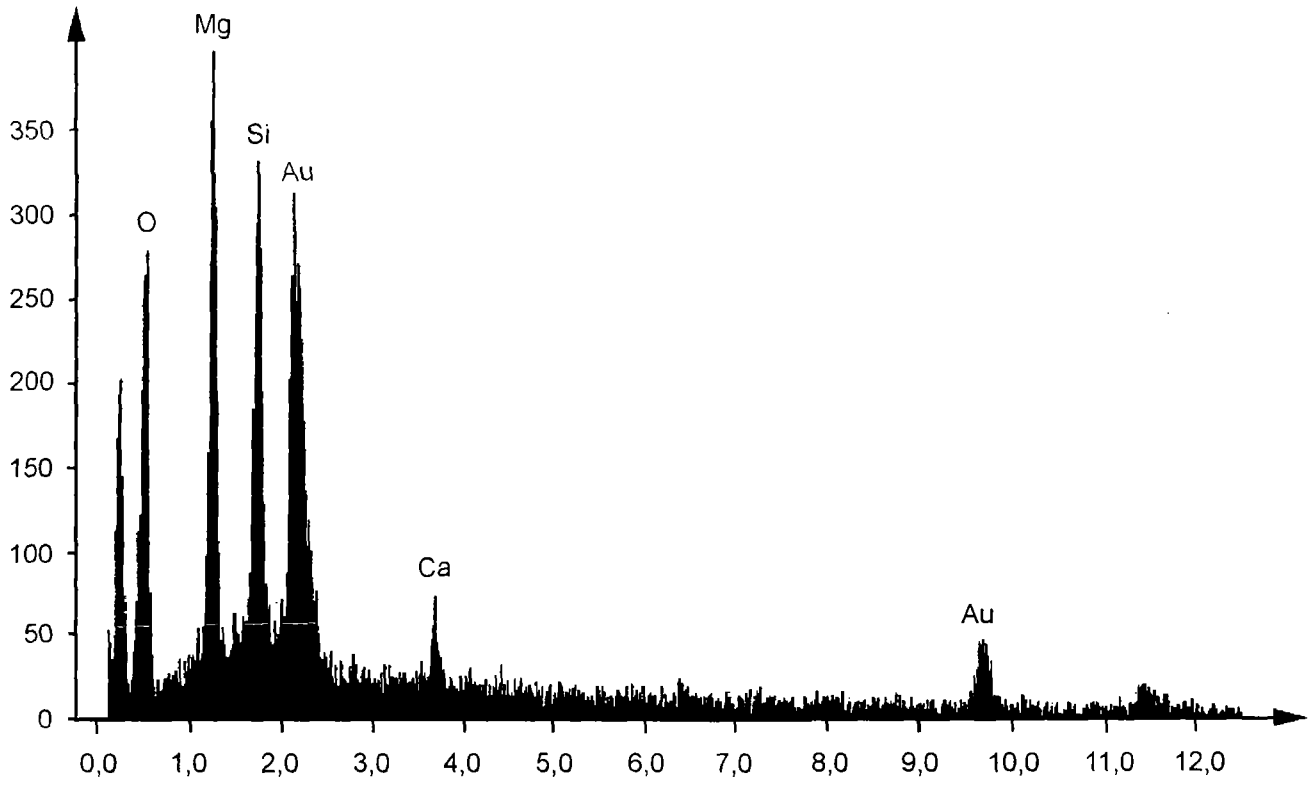


e) **Fibre of reference actinolite (no gold coating)**

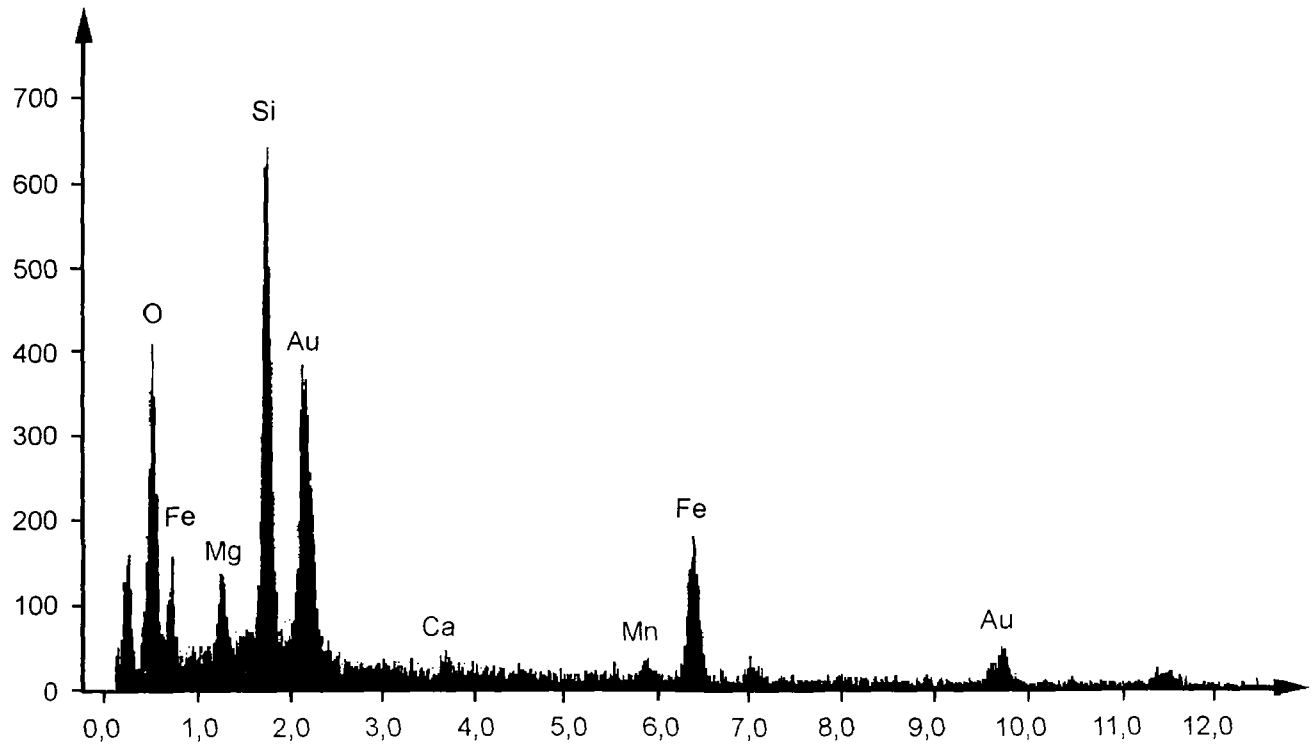


f) **Fibre of reference anthophyllite (no gold coating)**

Figure 8 (*continued*)

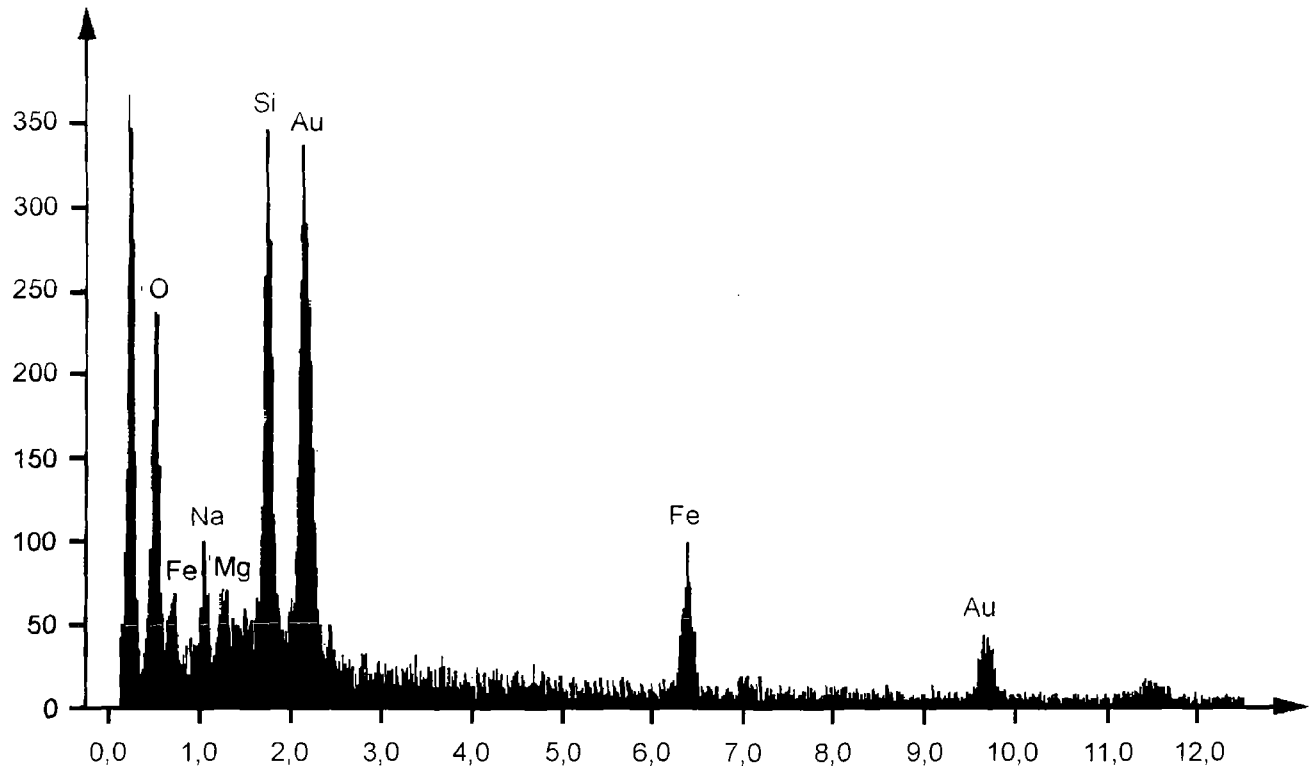


a) Fibre of chrysotile on a gold-coated filter with other particles



b) Fibre of amosite on a gold-coated filter with other particles

Figure 9 — Examples of EDXA spectra from asbestos fibres on gold-coated filters



c) Fibre of crocidolite on a gold-coated filter with other particles

NOTE The spectra were obtained from fibres detected in actual air samples.

Figure 9 (*continued*)

6.4.3.11 Precautions during acquisition of EDXA spectra

During the acquisition of EDXA spectra, care shall be taken to ensure that the electron beam is stable, that the point of incidence is on the fibre and that the beam does not drift off the fibre during the analysis. It is also necessary to ensure that the point of incidence of the electron beam is as far as possible from any attached or adjacent fibres and/or particles, in order to obtain a spectrum from the fibre with a minimum of interference.

In some cases, it is not possible to classify a fibre unambiguously. This could be because of interference by other particles or fibres, or because the peak to background ratios are insufficient. When this occurs, annotate the data for these fibres by an asterisk and indicate the reason on the fibre counting form.

6.4.3.12 EDXA spectra collected from actual air samples

Examples of EDXA spectra collected from chrysotile, amosite and crocidolite fibres detected in actual air samples are shown in Figure 9. Peaks from gold will always be present in these spectra, but their intensities will vary depending on the size of the fibre from which the spectrum is being acquired, and other factors relating to the interaction of the electron beam with the gold-coated substrate. Other peaks present in the spectra, and variations in relative peak intensities, can be attributed to adjacent or attached particles.

6.4.4 Measurement of fibre dimensions

For measurement of the fibre dimensions, particularly the fibre width, it is recommended to increase the magnification to about 10 000 × or higher. It is also recommended to acquire the EDXA spectrum at this magnification. Before moving the specimen stage and switching to a higher magnification, the position of a

prominent structure in the field of view should be noted, in order to allow correct repositioning of the specimen stage after analysis of the particular fibre has been completed.

6.4.5 Recording of data on the fibre counting form

For each fibre classified as "asbestos" or "other inorganic fibres", record the image field number, fibre number, length and width, elemental composition and fibre classification. On the fibre counting form, each fibre classified as "asbestos" shall be further classified as either "chrysotile" or "amphibole". It is recommended that the "amphibole" category be further divided, to the extent possible, into categories representing compositions consistent with the different varieties of amphibole asbestos.

Count calcium sulfate fibres, but do not measure their dimensions. Enter the count of calcium sulfate fibres on the fibre counting form as a tally list, as shown in Figure 5.

7 Calculation of results

7.1 Calculation of the mean fibre concentration

The measurement obtained by this method is the numerical concentration c_i (in fibres per cubic metre for fibre type i) of inorganic fibres longer than 5 µm, less than 3 µm in maximum width, and which also have minimum aspect ratios of 3:1. Based on the EXDA results, these fibres are classified into groups according to Table 1.

Table 1 — Fibre classifications

Fibre classification	Numerical concentration c_i
1 Fibres with compositions consistent with those of serpentine asbestos	c_1
2 Fibres with compositions consistent with those of amphibole asbestos	c_2
3 Other inorganic fibres	c_3
4 Calcium sulfate	c_4

Calculate the numerical concentration for fibre classification i (i = 1, 2, 3, 4) as follows:

$$c_i = \frac{n_i}{N \cdot V_B} \tag{3}$$

where

$$V_B = \frac{4 \cdot F_B \cdot V \times 10^6}{\pi \cdot d_{\text{eff}}^2} \tag{4}$$

and

c_i is the numerical fibre concentration of fibre classification i, in fibres per cubic metre;

n_i is the number of fibres counted for fibre classification i;

N is the number of image fields examined;

V_B is the sampled air volume, in cubic metres, per image field;

F_B is the area of the image field, in square millimetres;

V is the sampled air volume, in cubic metres;

d_{eff} is the effective filter diameter (diameter of the exposed circular filter area), in millimetres.

The data recorded on the fibre counting form can be used to determine the size distribution for fibres (except calcium sulfate fibres) with aspect ratios greater than 3:1 within the length range from 5 µm to 100 µm, and the width range from 0,2 µm to 3 µm.

Depending on the choice of sampling equipment, the sampled air volume is calculated as the difference between the meter readings of a gas-volume meter at the start and end of sampling, or from the average volume flowrate and sampling time.

The numerical concentration of fibres with chemical compositions consistent with those of the asbestos varieties is then calculated as:

$$c = c_1 + c_2 \tag{5}$$

and the concentration for all inorganic fibres C_T (excluding calcium sulfate fibres) is then calculated as:

$$c_T = c_1 + c_2 + c_3 \tag{6}$$

7.2 Calculation of the 95 % confidence interval

For the number of fibres n_i of classification i detected in the SEM examination, obtain the values for the lower and upper 95 % confidence limits, λ_L and λ_U, from Table 2. Convert these values into numerical fibre concentrations using the equations:

$$c_i^L = \frac{\lambda_L}{N \cdot V_B} \tag{7}$$

$$c_i^U = \frac{\lambda_U}{N \cdot V_B} \tag{8}$$

If n_i fibres of fibre classification i were counted during the examination, then there is a 95 % probability that the numerical fibre concentration will lie within this range.

8 Performance characteristics

8.1 General

Asbestos fibre concentrations measured in ambient air are generally of the order of less than 1 000 fibres/m^3, and mostly less than 100 fibres/m^3 (fibres longer than 5 µm). Consequently, the number of asbestos fibres counted during individual measurements is usually very low [12]. Calculation of the performance characteristics (measurement uncertainty, sampling uncertainty, analysis uncertainty) is therefore based on the total number of asbestos and other inorganic fibres.

8.2 Measurement uncertainty

8.2.1 Systematic errors

Systematic errors in the measured numerical fibre concentration can occur as a result of:

a) sampling (errors in measurement of volume flowrate);

b) SEM specimen preparation (fibre losses during handling and plasma ashing);

c) analysis (adjustment of SEM, fibre counting, measurement and identification).

The most critical items leading to systematic errors are those associated with the SEM examination. These include:

— detection and analysis of thin fibres with widths close to and lower than the calibrated visibility limit of 0,2 µm;

— subjective interpretation of aggregates comprising fibres and isometric particles during fibre counting;

— interpretation of EDXA spectra to classify fibres, particularly for spectra subject to interferences by coatings or adjacent particles.

8.2.2 Random errors

Random variation of the results occurs as a result of Poisson variability, and this is particularly important for low fibre counts. Low fibre counts are often experienced for asbestos fibres longer than 5 µm. The variability associated with particular fibre counts can be estimated using the Poisson distribution, as described in 8.2.6.

Where particular requirements are specified with regard to the accuracy of the measuring process, this statistical uncertainty in the results should always be taken into account during the planning of the sampling process and the evaluation of the extent of the measurement process resulting therefrom.

If extreme fluctuations in the fibre concentrations in ambient air occur as a result of, for example, meteorological influences, corresponding variations in the measurements will be found. In this type of situation, the sampling period and the number of individual measurements should be selected during planning of the air sampling in order to minimize the effects of such influences.

The errors described in 8.2.1 to 8.2.4 are defined as relative variables on the basis of experience, expressed as twice the standard deviation, which is approximately equivalent to a 95 % confidence interval. All of the errors specified relate to measurements of fibres longer than 5 µm and shorter than 100 µm.

8.2.3 Errors due to sampling

The sampling error is defined as the scatter of the measured results when using side-by-side identical sampling systems. On the basis of comparison measurements [17], the relative standard deviation was determined to be:

$$2\,\sigma_P < 15\ \%$$

8.2.4 Errors associated with the SEM examination

The errors associated with the SEM examination were calculated for the sums of fibres classified as asbestos and as other inorganic mineral fibres (excluding calcium sulfate fibres) from the results of four separate series of measurements in each of which there were five different participating laboratories [17]. From these results, a relative standard deviation of:

$$2\,\sigma_A \leqslant 35\ \%$$

was obtained.

From these comparison measurements [17], the relative standard deviation due to the subjective error of the operator, using one laboratory and one sample, was found to be:

$$2\,\sigma_{SF} = 15\,\%$$

This shows clearly that the equipment, subjective factors and the characteristics of the preparation are major aspects which determine the total error of the measurement.

8.2.5 Total error of the measurement

Provided that the errors from different sources are independent, the standard deviation for the overall measurement is given by:

$$\sigma_T = \sqrt{\sigma_P^2 + \sigma_A^2 + \sigma_S^2} \tag{9}$$

where

σ_T is the standard deviation for the overall measurement;

σ_S is the standard deviation for the sampling errors;

σ_A is the standard deviation for the analysis errors;

σ_P is the standard deviation for the Poisson variability.

The standard deviation for the combination of sampling and analysis is calculated from σ_P and σ_A to give:

$$2\sigma_V \leqslant 38\,\%$$

For individual samples, the standard deviation for the Poisson variability must be combined with these standard deviations to obtain the standard deviation for the measurement. The Poisson variability is dependent on the number of fibres counted, and is estimated as in 8.2.6.

8.2.6 Random errors due to fibre counting

Assuming that the numerical fibre concentrations on the filter are low, the probability P of detecting n fibres of a given fibre class during examination of N image fields can be described using the Poisson distribution:

$$P(n,a) = \frac{a^n \cdot \exp(-a)}{n!} \tag{10}$$

The variable a can be regarded as the product of the probability p of finding one fibre of the corresponding fibre class in one image field, and N, the number of image fields examined. It corresponds to the expected value n of the number of fibres detected during examination of N image fields. On the basis of the Poisson distribution, using Table 2, it is possible to determine the 95 % confidence interval for the calculated fibre number concentration.

For illustration, Figure 10 shows the probability density for detection of 0, 2 and 4 fibres in 165 image fields. The abscissa scale has been converted to fibre concentration in fibres per cubic metre, assuming a sampled air volume of 0,01 m^3 per image field.

Table 2 — Upper and lower limits of the Poissonian 95 % confidence interval of a count

Fibre count	Lower λ_L	Upper λ_U	Fibre count	Lower λ_L	Upper λ_U	Fibre count	Lower λ_L	Upper λ_U
0	0	3,689 [a]	46	33,678	61,358	92	74,164	112,83
1	0,025	5,572	47	34,534	62,501	93	75,061	113,94
2	0,242	7,225	48	35,392	63,642	94	75,959	115,04
3	0,619	8,767	49	36,251	64,781	95	76,858	116,14
4	1,090	10,242	50	37,112	65,919	96	77,757	117,24
5	1,624	11,669	51	37,973	67,056	97	78,657	118,34
6	2,202	13,060	52	38,837	68,192	98	79,557	119,44
7	2,814	14,423	53	39,701	69,326	99	80,458	120,53
8	3,454	15,764	54	40,567	70,459	100	81,360	121,66
9	4,115	17,085	55	41,433	71,591	110	90,400	132,61
10	4,795	18,391	56	42,301	72,721	120	99,490	143,52
11	5,491	19,683	57	43,171	73,851	130	108,61	154,39
12	6,201	20,962	58	44,041	74,979	140	117,77	165,23
13	6,922	22,231	59	44,912	76,106	150	126,96	176,04
14	7,654	23,490	60	45,785	77,232	160	136,17	186,83
15	8,396	24,741	61	46,658	78,357	170	145,41	197,59
16	9,146	25,983	62	47,533	79,482	180	154,66	208,33
17	9,904	27,219	63	48,409	80,605	190	163,94	219,05
18	10,668	28,448	64	49,286	81,727	200	173,24	229,75
19	11,440	29,671	65	50,164	82,848	210	182,56	240,43
20	12,217	30,889	66	51,042	83,969	220	191,89	251,10
21	13,000	32,101	67	51,922	85,088	230	201,24	261,75
22	13,788	33,309	68	52,803	86,207	240	210,60	272,39
23	14,581	34,512	69	53,685	87,324	250	219,97	283,01
24	15,378	35,711	70	54,567	88,441	260	229,36	293,62
25	16,178	36,905	71	55,451	89,557	270	238,75	304,23
26	16,983	38,097	72	56,335	90,673	280	248,16	314,82
27	17,793	39,284	73	57,220	91,787	290	257,58	325,39
28	18,606	40,468	74	58,106	92,901	300	267,01	335,96
29	19,422	41,649	75	58,993	94,014	310	276,45	346,52
30	20,241	42,827	76	59,880	95,126	320	285,90	357,08
31	21,063	44,002	77	60,768	96,237	330	295,36	367,62
32	21,888	45,175	78	61,657	97,348	340	304,82	378,15
33	22,715	46,345	79	62,547	98,458	350	314,29	388,68
34	23,545	47,512	80	63,437	99,567	360	323,77	399,20
35	24,378	48,677	81	64,328	100,68	370	333,26	409,71
36	25,213	49,840	82	65,219	101,79	380	342,75	420,22
37	26,050	51,000	83	66,111	102,90	390	352,25	430,72
38	26,890	52,158	84	67,003	104,00	400	361,76	441,21
39	27,732	53,315	85	67,897	105,11	410	371,27	451,69
40	28,575	54,469	86	68,790	106,21	420	380,79	462,18
41	29,421	55,622	87	69,684	107,32	430	390,32	472,65
42	30,269	56,772	88	70,579	108,42	440	399,85	483,12
43	31,119	57,921	89	71,474	109,53	450	409,38	493,58
44	31,970	59,068	90	72,370	110,63	460	418,92	504,04
45	32,823	60,214	91	73,267	111,73	470	428,47	514,50

[a] The one-sided upper 95 % confidence limit for zero structures is 2,99.

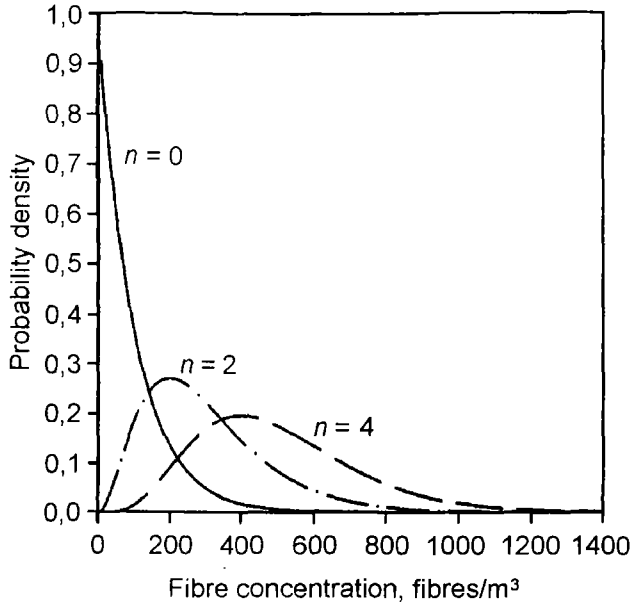


Figure 10 — Distribution of the probability density of the fibre concentration for 0, 2 and 4 fibres detected in an analysed air volume of 0,01 m^3

8.3 Limit of detection

The limit of detection is defined as the numerical fibre concentration below which, with 95 % confidence, the actual fibre concentration lies when no fibres are detected during the SEM examination.

The detection limit depends on:

a) the volume of air which passed through the filter during the period of sampling;

b) the effective area of the filter;

c) the area of filter examined.

If no fibres are detected during the SEM examination, Table 2 shows that the upper 95 % confidence limit is 2,99 fibres. The detection limit, E, for the measurement is given by:

$$E = \frac{2,99}{N \cdot V_B} \tag{11}$$

A limit of detection of approximately 300 fibres/m^3 is obtained with a sampled air volume of 1 m^3 per square centimetre of filter surface area, if an area of 1 mm^2 is examined in the SEM. This corresponds to evaluation of an air volume of 0,01 m^3.

Theoretically, the limit of detection can be reduced indefinitely by increasing the area of filter examined in the SEM. For example, the limit of detection can be reduced to approximately 150 fibres per cubic metre if, with a sampled air volume of 1 m^3 per square centimetre of filter area, the SEM examination is extended to an area of 2 mm^2.

Any background contamination by fibres which may exist on unused filters is not taken into account in the quoted detection limit. Experience has shown, however, that background contamination levels of unused filters are negligible compared with the above detection limit.

The limit of detection may also be reduced by increasing the volume of air sampled. However, the extent to which the sampled air volume can be increased may be limited by the concentration of non-fibrous particles in the ambient air. This can result in an increased formation of agglomerates on the filter and consequent obscuration of fibres, or the pores of the filter may become blocked during sampling, leading to an unacceptable increase in the differential pressure across the filter.

The choice of an appropriate sampling time and the necessary extent of the SEM examination are therefore aspects of the measurement which shall be optimized at the planning stage for each particular application.

9 Test report

The test report shall include at least the following information:

a) reference to this International Standard;

b) identification of the sample;

c) the date, time and location of the air sample collection, and all necessary sampling data, including the volume of air sampled, the sampling time and the effective diameter of the sample collection filter;

d) the analysed air volume;

e) the analytical sensitivity;

f) the number of fibres in each of the fibre classifications detected during the SEM examination, and their calculated numerical concentrations.

It is recommended that the upper and lower 95 % confidence limits be reported for the fibres classified as asbestos and those classified as other inorganic fibres. An example of a test report is shown in Figure 11.

TEST REPORT

"Project title"

Sampling	
Date: "*Date of sample collection*"	Sample volume: 3,8 m^3
Measurement number: "*Number*"	Sampling duration: 8 h
Sampling apparatus: "*Type*"	Effective filter area: 380 mm^2
Type of measurement: "....."	
Location: "*Description of the sampling location*"	
Air sampling by:	

Analysis in the SEM			
Scanning electron microscope: "*Type of SEM*"			
Analysis system: "*Type of EDXA*"		Magnification: 2 000 ×	
Filter area analysed: 1 mm^2		Number of image fields examined: 100	
Image field area: 0,01 mm^2		Number of image fields rejected: 5	
Number of rejected:	Bundles: 1	Clusters: 0	Matrices: 2
Number of fibres without spectrum: 4		Micrograph Numbers: 456, 457, 458	

Results			
Fibre classification	**Number of fibres counted**	**Fibre concentration (fibres/m^3)**	
		Mean	**Poissonian 95 % confidence interval**
Amphibole	6	600	220 to 1 310
Chrysotile	3	300	60 to 880
Total asbestos	9	900	410 to 1 710
Other inorganic	15	1 500	840 to 2 470
Calcium sulfate	8	800	— —

Sampling and analysis performed in accordance with ISO 14966.

Detection of one fibre corresponds to a concentration of 100 fibres/m^3.

The volume of air analysed was 0,01 m^3.

Figure 11 — Example of a test report

Annex A
(normative)

Preparation of filters for air sampling

The membrane filters of a new batch shall be examined in the SEM to establish that any background level of inorganic fibres is sufficiently low that it does not significantly affect the reported results, and that the filters have a uniform pore distribution.

Before sampling, it is necessary to coat the surface of the membrane filters with a gold layer. The gold coating protects the filter during plasma ashing and allows the filter to be examined in the SEM without the increase in the geometric dimensions of the inorganic fibres, which would result if an evaporated film were applied after sampling. The gold coating is applied using either a vacuum evaporator or a sputter-coating unit.

The thickness of the gold coating applied to the side of the filter on which particles are to be collected during air sampling (the smooth and more strongly reflecting side) shall be approximately 30 nm. A uniform thickness of gold coating is required in order to minimize variations in contrast in the SEM image. Optional vacuum deposition of approximately 20 nm thickness of gold onto the other side of the filter protects the sampling filter during ashing and can help to improve the contrast of fibres in the SEM image. If a means of measuring the thickness of the gold coating is not available in the vacuum evaporator, the gold coating can be assumed to be satisfactory when the filter loses its initial dark colour during the course of the evaporation and takes on a typical metallic gold lustre. If, in addition, a coated filter appears to have a green colour when observed in transmitted light, it can be assumed that the thickness of the gold coating is within the required limits.

The filter coating thickness can also be checked easily using the SEM. A number of membrane filters are initially weighed, after which they are coated with gold and weighed again. The mass of the gold coating can be calculated by subtraction, and the thickness can be determined from the area of the filters and the density of gold. Using a constant beam current in the SEM, the height or peak integral for one of the gold X-ray peaks produced by these reference filters can then be compared with the corresponding gold peak produced on a filter prepared for air sampling. The thickness of the gold coating on the sampling filter can be calculated, assuming a linear relationship between the gold coating thickness and the size of the gold peak. After subtracting the background signal, the integral under the gold peak, or the height of this peak, is a direct measure of the thickness of the gold coating.

Annex B
(normative)

Procedures for calibration and adjustment of the SEM

B.1 Calibration of the scanning electron microscope

The SEM specimen is examined at an accelerating voltage of approximately 20 kV and a magnification of between 2 000 × and 2 500 ×. For fibre identification in the SEM, an accelerating voltage of 20 kV is recommended.

The magnification on the screen shall be calibrated using a certified commercially-available magnification standard. It is important to recognize that the magnification value displayed on some models of SEM is that applicable to micrographs produced by the recording system, and not to the viewing screen [cathode ray tube (CRT) display]. The SEM examination is performed directly on the viewing screen, and the magnification calibration must relate to the viewing screen.

Adjust the SEM in such a way that chrysotile fibres with a width of 0,2 µm and lengths between 5 µm and 10 µm are visible at a the counting magnification of approximately 2 000 ×. This adjustment is performed by selecting a fibre, either on the prepared sample or on a test sample, which is just visible at the magnification used for counting. The width of this fibre is then determined at a magnification of approximately 20 000 ×. This adjustment shall be carried out on at least two separate fibres before starting the analysis, and shall be repeated several times during the course of a series of analyses.

NOTE 1 On a 30 cm CRT display, 100 image fields at a magnification of approximately 2 000 × correspond to an area of approximately 1 mm^2 on the specimen.

NOTE 2 The width of the scan line (or pixel width for an SEM with digital imaging) on the sample and the diameter of the electron beam are the factors which determine the resolution in the SEM. Provided that the scan line width or the pixel width does not exceed 0,2 µm, no severe image degradation relevant to resolution of a 0,2 µm wide fibre longer than 5 µm is observed. With currently-available CRT display sizes and nominal line numbers of about 500 to 700 at magnifications of 2 000 × or 2 500 ×, the above conditions are usually met.

B.2 Adjustment of the EDXA system

The largest possible solid angle of the EDXA detector system should be used. The operating parameters of the SEM and the X-ray detector system shall be selected so that a statistically-acceptable X-ray spectrum can be acquired from a 0,2 µm width chrysotile fibre on the test sample within a maximum period of 100 s.

The criterion for statistical acceptability requires, for peak height, P, and background level, B:

$$P > 3\sqrt{B} \tag{B.1}$$

with a minimum of 30 pulses in the channel corresponding to the maximum peak height for each of the magnesium and silicon peaks [11]; and

$$\frac{P+B}{B} > 2 \tag{B.2}$$

for each of the magnesium and silicon peaks.

B.3 Adjustment of the plasma asher

The operating conditions of the plasma asher shall be adjusted so that the time for removal of organic materials from sample filters can be predicted. Place a burning candle which produces smoke at a distance of approximately 30 cm from a sampling head equipped with a gold-coated filter in accordance with annex A. Using the nominal volume flowrate, collect smoke on the filter for approximately 30 s. Remove the filter from the sampling head (it should be greyish or black at the surface) and place it in the plasma asher. Cover approximately half of the filter surface with a glass microscope slide.

If the ashing conditions are correctly adjusted, the smoke particles from the unprotected half of the filter will be removed in approximately 20 min. The colour of this filter area should then appear the same as that of the unused filter. The filter material itself should appear unchanged.

B.4 Detection of leaks in the sampling head

The method for collecting smoke as described in B.3 can also be used to determine whether there are leaks around the filter when it is installed in the sampling head. After exposure of the sampling head to the smoke, a leak will result in colouration around the edge of the backing filter or around the edge of the gold-coated filter. If this occurs, improve the fitting of the filter in the sampling head, so that an airtight seal is obtained.

Annex C
(informative)

Characteristics and chemical composition of inorganic fibres

C.1 General

For the purposes of this International Standard, particles with a minimum ratio of length to width of 3:1 are defined as fibres.

All fibrous particles can be classified according to their chemical composition (inorganic/organic), according to their physical structure (amorphous/crystalline) and according to their origin (natural/synthetic) [18]. The term "synthetic inorganic fibres" is often used as a synonym for "artificial mineral fibres", or "man-made mineral fibres".

The development of fibrous morphology in a material can be attributed to a number of causes.

a) The lattice structure of a crystalline material may contain privileged directions such as lattice planes, lattice constants, or zones, which demand a fibrous habit. The chain or band silicates, such as pyroxenes, amphiboles and wollastonite, or the sheet silicates such as chrysotile, which have Si_4O_{10} sheets with a polar structure whose free valences point in the same direction, are examples of these types of structure [18 to 20].

b) The physical and chemical conditions during formation, such as pressure, temperature and the chemical species in solution, can lead to growth in a fibrous habit:

   1) monocrystalline "whiskers", such as tin, alumina, silicon carbide and potassium titanate, can be formed by crystallization from solution;

   2) quartz and aragonite can be formed by crystallization from a gel phase;

   3) alkaline sulfates can be produced during the formation of druses and pores in bricks, and;

   4) chrysotile asbestos, amosite, crocidolite and fibrous gypsum can be formed during metamorphosis of rock [19, 20].

c) Amorphous fibres can be manufactured from numerous inorganic mixtures. In these processes, the material is solidified from a melt or a solution process, using procedures such as spinning or stretching to form fibres such as glass fibres. After formation, the properties or crystallinity are sometimes modified by thermal treatment.

C.2 Asbestos fibres

Asbestos is a term used to designate a group of naturally occurring inorganic crystalline silicates when they occur in a fibrous habit. They occur as individual fibres, or bundles of fibres, in rocks. During transformation of the rock, growth of fibres is favoured by conditions in fissures and gaps in the rock [19, 20].

The asbestos types can be classified mineralogically into serpentine asbestos (chrysotile asbestos) and amphibole asbestos [19 to 23]. Serpentine asbestos forms during hydrothermal transformation of ultra-basic rocks containing magnesium and aluminium, such as dunites, gabbros and basalts, at temperatures lower than 360°C. The starting minerals are olivine and lower-ranking pyroxenes and amphiboles. If whole rock complexes are involved in this transformation process (serpentinization), the final product is referred to as serpentine. During further hydrothermal mobilization, chrysotile fibre bundles may precipitate in fissures and gaps of the serpentine, with an intermediate gel phase.

Although chrysotile is a sheet silicate mineral, stresses in the lattice structure cause curvature in the sheet, which results in the formation of spiral scrolls and a fibrous habit. The diameter of these scrolls (fibrils) is variable and in the range of 0,02 µm to 0,05 µm. It is therefore possible to separate macroscopic chrysotile fibre bundles into fundamental fibrils of these diameters.

Asbestos varieties of the amphibole minerals also result from rock metamorphoses [19 to 23]. The amphiboles are chain silicates which exhibit a preferred crystallographic direction, which is apparent from the crystal morphology. Amphibole minerals appear to be columnar, acicular or fibrous, and they exhibit prominent cleavage parallel to the longitudinal axis of the fibre.

The ranges of composition for the various asbestos types are shown in Table C.1. The compositions include oxides of elements which do not necessarily appear in the nominal formulae. Elements other than those of the nominal formulae can occupy certain lattice points in the structure. Aluminium, for example, can substitute silicon in some lattice positions, and iron, manganese, titanium, nickel, chromium, lithium or zinc can substitute calcium or magnesium. Substitution of iron and magnesium by other elements is common.

Table C.1 — Chemical composition of various asbestos types (% mass fraction)

	Chrysotile	Amosite	Crocidolite	Anthophyllite	Tremolite	Actinolite
SiO_2	36 to 44	49 to 53	49 to 56	53 to 60	55 to 60	51 to 56
MgO	38 to 42	1 to 7	0 to 3	17 to 34	20 to 26	12 to 20
FeO	0 to 3	34 to 44	13 to 21	0 to 20	0 to 5	5 to 15
Fe_2O_3	0 to 5	0 to 5	13 to 20	0 to 5	0 to 5	0 to 5
Al_2O_3	0 to 2	0 to 1	0 to 1	0 to 3	0 to 3	0 to 3
CaO	0 to 2	0 to 2	0 to 3	0 to 3	10 to 15	10 to 13
K_2O	0 to 1	0 to 1	0 to 1	0 to 1	0 to 1	0 to 1
Na_2O	0 to 1	0 to 1	4 to 9	0 to 1	0 to 2	0 to 2
H_2O	12 to 14	2 to 5	2 to 5	1 to 6	1 to 3	1 to 3

C.3 Other fibrous minerals

C.3.1 General

A large number of natural minerals exist which, due to their lattice structure or the particular conditions of formation, crystallize in a fibrous habit. A number of these minerals which also occur in technical processes and as constituents in their products are listed here. For the many other such minerals, reference should be made to the relevant mineralogical and petrographical literature [19, 20, 22].

C.3.2 Silicates

Mullite $Al_6Si_2O_{13}$ and willemite Zn_2SiO_4 occur widely as fibrous, acicular or columnar individual crystals or as felt-like, partly spherulitic crystallite bundles in mineral phases in stoneware clays, porcelain, chamotte, and in silica and high alumina refractory bricks after use in zinc smelting plants and open-hearth furnaces.

Sillimanite Al_2SiO_5 and dumortierite $Al_7O_3(BO_3)(SiO_4)_3$, as fibrous varieties, are found only in the raw materials used for the production of high alumina refractory bricks [19, 24].

Pseudo-wollastonite $CaSiO_3$ is found as a columnar fibrous form in ceramic clays, iron blast furnace slags, welding electrodes and as devitrifying elements in soda-lime glass [19, 24].

Numerous members of the zeolite group, particularly wide-meshed tectosilicates with ring systems of SiO_4, or AlO_4 tetrahedra linked by channel-like elements, are fibrous. The channels in the zeolite crystal structures give rise to reversible ion exchange, molecular sieve and hydration properties. They are used as catalysts in cracking of petroleum, and in isomerization.

Important fibrous zeolites include the mixed crystals natrolite and scolecite $Na_2Al_2Si_3O_{10}{\cdot}2H_2O$ to $CaAl_2Si_3O_{10}{\cdot}3H_2O$. The cubic zeolites faujasite $(Na_2,Ca)Al_2Si_4O_{12}{\cdot}6H_2O$, erionite $(Ca,Na_2,K_2)_{1,5}$–$(Al_9Si_{27}O_{72}){\cdot}27H_2O$ and mordenite $(Ca,K_2,Na_2)Al_2Si_{10}O_{24}{\cdot}7H_2O$ frequently exhibit a fibrous habit. Industrially synthesized zeolites are usually used, since it is then possible to control the material properties more precisely [19].

C.3.3 Sulfates

Fibrous sulfates are common. Anhydrite $CaSO_4$ is found in mortar, in concrete after exposure to sulfuric acid and sulfate solutions, and in fly ash. Gypsum $CaSO_4{\cdot}2H_2O$ is found in plaster of Paris and sculptor's plaster after it is mixed with water, and it is also found in other building materials. Ettringite $Ca_6Al_2(SO_4)_3(OH)_{12}{\cdot}24H_2O$ occurs as a binding agent in high-alumina cements. Hydrated tricalcium aluminate in these cements reacts with sulfate solutions to form bundles of acicular ettringite fibres, as new formations in hydrated brown coal flyash and as satin white filler pigments in paper.

Epsomite $MgSO_4{\cdot}7H_2O$ and mirabilite $Na_2SO_4{\cdot}10H_2O$ occur as "blooms" in soil, and the latter also in masonry.

C.3.4 Industrial crystalline fibres

The demand for fibre-reinforced high-performance composite materials is growing continuously. Since the properties of natural fibres do not satisfy industrial requirements, a large number of synthetic inorganic fibres have been developed.

These fibres are either polycrystalline, each consisting of a large number of smaller fibrous crystals oriented in the same direction, or each is single crystal whisker which has grown into a fibre as a result of a privileged direction, generally a helical dislocation.

The polycrystalline fibres generally exhibit a columnar form with a finely structured surface similar to that of the source material used for manufacture. Monocrystalline fibres (whiskers) have a primarily plane form with a polygonal cross-section. Examples of synthetic inorganic crystalline fibres are shown in Table C.2 [25 to 27].

C.3.5 Amorphous fibres

Amorphous inorganic fibres, also referred to as "man-made vitreous fibres", are produced by a melting process. The primary constituents of the melt are silicates, as shown by the compositional ranges in Table C.3. Table C.4 shows typical fibre diameters and a classification according to the type of raw materials used [25, 27].

The strands of melted material, which are cooled at various rates, solidify to form glassy fibres. The fibres generally exhibit a circular cross-section and a structureless, smooth surface.

The mechanical drawing process used for production of textile glass fibres generally yields fibres which have a constant diameter over their full length. Non-textile glass fibres, manufactured by processes such as centrifugal blowing, can have irregular fibre thicknesses with droplet-like thickenings, but these materials may also contain very fine fibres with diameters lower than 1 µm. Non-textile glass fibres include glass wool, cinder wool, rock wool and ceramic wools [25].

Table C.2 — Synthetic inorganic crystalline fibres

	Fibre diameter µm	Morphology	Method of manufacture
Carbon/graphite	5 to 10	columnar	microfibrillation of oriented polyacrylonitrile or cellulose fibres by carbonization or graphitization
Boron	100	columnar	precipitation of boron as a "corn cob" structure on 12 µm diameter tungsten core
Boron carbide (B_4C)	1 to 25	planar	reaction of primary carbon fibre with boron trichloride and hydrogen
Boron nitride (BN)	4 to 8		reaction of primary B_2O_3 fibre with ammonia
Alumina (α-Al_2O_3)	3 to 9		formation of α-Al_2O_3 from a polymer gel phase in nitrogen at 100°C
Silicon carbide (SiC)	2 to 8		SiO_2-impregnated cellulose fibres are pyrolysed and converted to SiC in a protective gas atmosphere
Zirconia (ZrO_2)	3 to 6	columnar	cellulose fibres impregnated with a zirconium salt are pyrolysed and the carbon is removed
Tungsten	12		primarily sintering processes
Steel (austenitic)	12		bundle drawing process with ductile matrix (copper) or melt-drawing process in a glass matrix
α-Alumina (α-Al_2O_3)	0,5 to 10	planar	precipitation from a gas phase
α-Alumina (α-Al_2O_3)	50 to 100	columnar	melt-drawing process
α-SiC	0,5 to 10		precipitation from the vapour phase onto finely dispersed lanthanum
β-SiC	0,5 to 10		decomposition of methyltrichlorsilane in hydrogen and precipitation onto carbon
Potassium hexatitanate $K_2Ti_6O_{13}$	0,2 to 1		melt crystallization

Table C.3 — Compositions of synthetic inorganic amorphous fibres

Component	Textile glass	Insulating glass	Rock fibres	Slag fibres	Ceramic fibres
SiO_2	54 to 74	48 to 63	45 to 53	≈ 41	45 to 52
Al_2O_3	0 to 25	3 to 9	6 to 13	≈ 12	42 to 51
B_2O_3	0 to 22	0 to 6			0 to 1
Fe_2O_3	0 to 5	3 to 14	5 to 8		1 to 16
FeO		0 to 4	1 to 7	≈ 1	
CaO	0 to 17	7 to 28	11 to 30	37 to 40	0 to 3
MgO	0 to 6	32 to 38	3 to 10	≈ 4	0 to 5
BaO	0 to 1	0 to 25			
ZrO_2	0 to 16				0 to 3
TiO_2	0 to 2	0 to 2	5 to 2	≈ 0,4	0 to 6
MnO		0 to 5	6 to 5	≈ 0,5	
P_2O_5		0 to 6	0 to 1	≈ 0,3	
CaS				0 to 1	
Li_2O	0 to 1				
K_2O	0 to 8	8 to 36	≈ 1,3	≈ 0,4	0 to 3
Na_2O	0 to 13	4 to 14	≈ 2,4	2 to 14	0 to 7
S				≈ 0,4	
F				≈ 0,4	

Table C.4 — Raw materials used in the manufacture of synthetic inorganic, amorphous fibres

Fibre	Diameter μm	Source
Glass fibres	0,15 to 35	Natural minerals or rocks
Stone fibres	0,3 to 14	Natural minerals or rocks
Slag fibres	0,4 to 5	Metallurgical or non-metallurgical slags
Ceramic fibres	0,2 to 10	Melts primarily of alumina and silica
Quartz fibres	1 to 10	Pure quartz melts
Silica glass fibres	< 5 to 10	Leaching and thermal treatment of aluminium borosilicate glass

Annex D
(informative)

Poisson variability as a function of fibre density on sampling filter and area of filter analysed

Figure D.1 shows the relationship between the fibre density on the filter, the area of filter examined by the analyst, and the resulting unavoidable statistical fluctuations in the number of fibres counted. Recognition of these limitations are important in design of the sampling. The standard deviations shown in the graphs are calculated on the assumption that the distribution of fibres on the filter is Poissonian, for which the standard deviation of a fibre count is the square root of the fibre count. The variability is shown as twice the relative standard deviation, σ_s. The 95 % confidence intervals for the Poisson distribution are asymmetric about the mean, particularly for low numbers of fibres. The 95 % confidence intervals are shown in Table 2.

The curves illustrate the requirement to consider variability of fibre counting in design of strategies for sample collection. For example, assuming that the fibres are distributed on the filter according to a Poisson distribution, for a fibre density of 20 fibres per square millimetre of filter area the variability of the fibre count, expressed as $2\sigma_s$ after the examination of a filter area of 0,7 mm^2, is shown to be approximately 50 %. For lower fibre densities, analysis of larger areas of filter is necessary in order to achieve a comparable variability of approximately 50 %. Correspondingly, if higher fibre densities can be obtained on the filters, either because a higher airborne fibre concentration exists or because the suspended particulate is sufficiently low that the sampling time can be extended, the area of filter examined can be reduced, while still maintaining an acceptable variability for the measurement. In some cases, a combination of the two approaches can be an expedient means of improving the precision of the measurements.

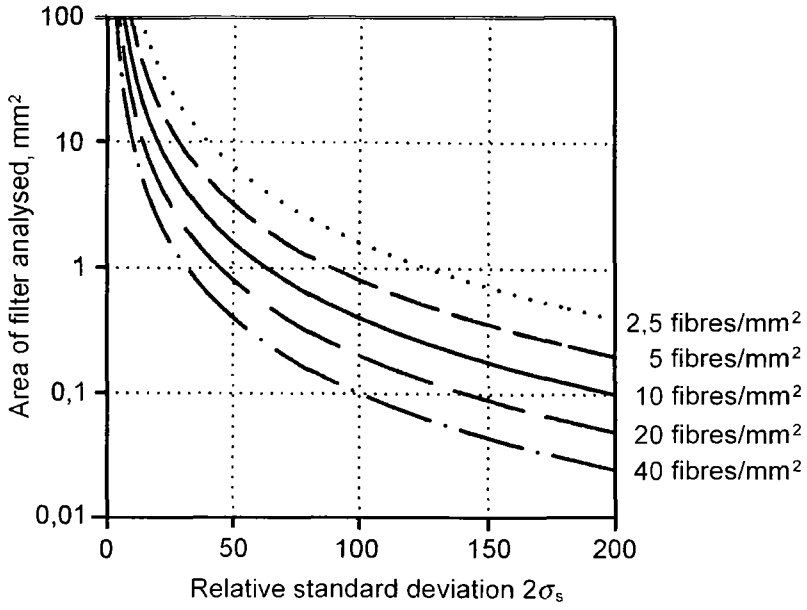


Figure D.1 — Poisson variability of fibre counts as a function of fibre density on the sampling filter and area of filter analysed

Annex E
(informative)

Combination of the results from multiple samples

For some applications, for example to reduce the error in the measured result, it may be desirable to calculate a mean value from several samples. A mean air concentration for the same location, but over a longer period of time, can also be derived by combining the results from a number of air samples collected sequentially over the required time period.

The measured fibre concentration is determined by two parameters: the number of fibres counted during the SEM examination and the volume of air examined by the analyst. The detection limit is determined solely by the volume of air examined by the analyst.

A mean concentration, $\overline{c_i}$, for fibres of type i is derived by summation of the fibres from all contributing samples and dividing by the summation of the individual volumes analysed in each sample.

$$\overline{c_i} = \frac{\sum n_i}{\sum V_P} \tag{E.1}$$

The best estimate for the mean concentration is given by:

$$\langle \overline{c_i} \rangle = \frac{\sum n_i + 1}{\sum V_P} \tag{E.2}$$

The detection limit, E, for the mean concentration is:

$$E = \frac{2{,}99}{\sum V_P} \tag{E.3}$$

for the mean fibre concentration, the 95 % confidence interval due to Poisson variability is calculated on the basis of n_i and V_P.

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Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Mass Concentration[1]

This standard is issued under the fixed designation D 5756; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure to (*a*) identify asbestos in dust and (*b*) provide an extimate of the concentration of asbestos in the sampled dust, reported as either the mass of asbestos per unit area of sampled suurface or as the mass of asbestos per mass of sampled dust.

1.1.1 If an estimate of asbestos structure counts is to be determined, the user is referred to Test Method D 5755.

1.2 This test method describes the equipment and procedures necessary for sampling, by a microvacuum technique, non-airborne dust for levels of asbestos. The non-airborne sample is collected inside a standard filter membrane cassette from the sampling of a surface area for dust which may contain asbestos.

1.2.1 This procedure uses a microvacuuming sampling technique. The collection efficiency of this technique is unknown. Variability of collection efficiency for any particular substrate and across different types of substrates is also unknown. The effects of sampling efficiency differences and variability on the interpretation of dust sampling measurements have not been determined.

1.3 Asbestos identified by transmission electron microscopy (TEM) is based on morphology, selected area electron diffraction (SAED), and energy dispersive X-ray analysis (EDXA). Some information about structure size is also determined.

1.4 This test method is generally applicable for an estimate of the concentration of asbestos starting from approximately 0.24 pg of asbestos per square centimeter (assuming a minimum fiber dimension of 0.5 μm by 0.025 μm, see 17.8), but will vary with the analytical parameters noted in 17.8.

1.4.1 The procedure outlined in this test method employs an indirect sample preparation technique. It is intended to disaggregate and disperse asbestos into fibrils and fiber bundles that can be more accurately identified, counted, and sized by transmission electron microscopy. However, as with all indirect sample preparation techniques, the asbestos observed for quantitation may not represent the physical form of the asbestos as sampled. More specifically, the procedure described nmeither creates not destroys asbestos, but it may alter the physical form of the mineral fibers.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 1739 Test Methods for Collection and Measurement of Dustfall (Settleable Particulate Matter)[3]

D 3195 Practice for Rotameter Calibration[3]

D 5755 Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations[3]

E 832 Specification for Laboratory Filter Papers[4]

2.2 *ISO Standards:*

ISO/10312 Ambient Air: Determination of Asbestos Fibers; Direct Transfer Transmission Electron Microscopy Procedure[5]

ISO/CD13794 Ambient Air: Determination of Asbestos Fibres; Indirect-Transfer Transmission Electron Microscopy Procedure[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *asbestiform*—a special type of fibrous habit in which the fibers are separable into thinner fibers and ultimately into fibrils. This habit accounts for greater flexibility and higher tensile strength than other habits of the same mineral. For more

[1] This test method is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheresand is the direct responsibility of Subcommittee D22.07on Sampling and Analysis of Asbestos.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.03.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

information on aasbestiform mineralogy, see references **(1)**, **(2)** and **(3)**.[6]

3.1.2 *asbestos*—a collective term that describes a group of naturally occurring, inorganic, highly fibrous silicate minerals, which are easily separated into long, thin, flexible fibers when crushed or processed.

3.1.2.1 *Discussion*—Included in the definition are the asbestiform varieties of: serpentine (chyrsotile); riebeckite (crocidolite); grunerite (amosite); anthophyllite (anthophyllite asbestos); tremolite (tremolite asbestos); and actinolite (actinolite asbestos). The amphibole mineral compositions are defined according to the nomemclature of the International Mineralogical Association **(3)**.

Asbestos	Chemical Abstract Service No.[7]
Chrysotile	12001-29-5
Crocidolite	12001-28-4
Grunerite Asbestos (Amosite)	12172-73-5
Anthophyllite Asbestos	77536-67-5
Tremolite Asbestos	77536-68-6
Actinolite Asbestos	77536-66-4

3.1.3 *fibril*—a single fiber that cannot be separated into smaller components without losing its fibrous properties or appearance.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aspect ratio*—the ratio of the length of a fibrous particle to its average width.

3.2.2 *bundle*—a structure composed of three or more fibers in a parallel arrangement with the fibers closer than one fiber diameter to each other.

3.2.3 *cluster*—an aggregate of two or more randomly oriented fibers, with or without bundles. Clusters occur as two varieties—disperse clusters and compact clusters.

3.2.3.1 *compact cluster*—a complex and tightly bound network in which one or both ends of each individual fiber or bundle are obscured, such that the dimensions of individual fibers or bundles cannot be unambiguously measured.

3.2.3.2 *disperse cluster*—a disperse and open network in which both ends of one of the individual fibers or bundles can be separately located and its dimensions measured.

3.2.4 *debris*—materials that are of an amount and size (particles greater than 1 mm in diameter as defined by a 1.0 by 1.0 mm screen) that can be visually identified (by color, texture, etc.) as to their source.

3.2.5 *dust*—any material composed of particles in a size range of ≤1 mm and large enough to settle by virtue of their weight from the ambient air. See Test Method D 1739.

3.2.6 *fiber*—a structure having a minimum length of 0.5μ m with an aspect ratio of 5 to 1 or greater and substantially parallel sides **(4)**. Fibers are assumed to have a cylindrical shape **(5)**.

3.2.7 *fibrous mineral*—a mineral that is composed of parallel, radiating, or interlaced aggregates of fibers, from which the fibers are sometimes separable.

3.2.7.1 *Discussion*—The crystalline aggregate may be referred to as fibrous even if it is not composed of separable fibers, but has that distinct appearance. The term fibrous is used in a general mineralogical way to describe aggregates of grains that crystallize in a needle-like habit and appear to be composed of fibers. Fibrous has a much more general meaning than asbestos. While it is correct that all asbestos minerals are fibrous, not all minerals having fibrous habits are asbestos.

3.2.8 *indirect preparation*—a method in which a sample passes through one or more intermediate steps prior to final filtration.

3.2.9 *matrix*—a structure in which one or more fibers, or fiber bundles, touch, are attached to, or partially concealed by a single particle or connected group of non-fibrous particles. The exposed fiber must meet the fiber definition (see section 3.2.6). Matrices occur as two varieties—disperse matrices and compact matrices.

3.2.9.1 *compact matrix*—a structure consisting of a particle or linked group of particles, in which fibers or bundles can be seen either within the structure or projecting from it, such that the dimensions of individual fibers and bundles cannot be unambiguously determined.

3.2.9.2 *disperse matrix*—a structure consisting of a particle or linked group of particles, with overlapping or attached fibers or bundles in which at least one of the individual fibers or bundles can be separately identified and its dimensions measured.

3.2.10 *structures*—a term that is used to categorize all the types of asbestos particles which are recorded during the analysis (such as fibers, bundles, clusters, and matrices).

4. Summary of Test Method

4.1 The sample is collected by vacuuming a known surface area with a standard 25 or 37 mm air sampling cassette using a plastic tube that is attached to the inlet orifice which acts as a nozzle. The sample is transferred from inside the cassette to a 50/50 alcohol/water solution and screened through a 1.0 by 1.0 mm screen. The fine dust is filtered onto a membrane filter and ashed in a muffle furnace. The ash is mixed with distilled water to a known volume. Aliquots of the suspension are then filtered through a membrane. A section of the membrane is prepared and transferred to a TEM grid using the direct transfer method. The asbestiform structures are identified, sized, and counted by TEM, using SAED and EDXA at a magnification dependent on the size range of asbestos structures present.

5. Significance and Use

5.1 This microvacuum sampling and indirect analysis method is used for the general testing of non-airborne dust samples for asbestos. It is used to assist in the evaluation of dust that may be found on surfaces in buildings, such as ceiling tiles, shelving, electrical components, duct work, carpet, etc. This test method provides an estimate of the mass concentration of asbestos in the dust reported as either the mass of asbestos per unit area or as the mass of asbestos per mass of sampled dust as derived from a quantitative TEM analysis.

5.1.1 This test method does not describe procedures or techniques required to evaluate the safety or habitability of buildings with asbestos-containing materials, or compliance with federal, state, or local regulations or statutes. It is the

[6] The boldface numbers refer to the list of references at the end of the test method.

[7] The non-asbestiform variations of the minerals indicated in 3.1.2 have different Chemical Abstract Service (CAS) numbers.

user's responsibility to make these determinations.

5.1.2 At present, a single direct relationship between asbestos-containing dust and potential human exposure does not exist. Accordingly, the user should consider these data in relationship to other available information in their evaluation.

5.2 This test method uses the definition *settleable particulate matter* found in Test Method D 1739 as the definition of dust. This definition accepts all particles small enough to pass through a 1 mm screen. Thus, a single, large asbestos-containing particle(s) (from the large end of the particle size distribution) disassembled during sample preparation may result in anomalously large asbestos concentration results in the TEM analyses of that sample. Conversely, failure to disaggregate large particles may result in anomalously low asbestos mass concentrations. It is, therefore, recommended that multiple independent samples be secured from the same area, and that a minimum of three samples be analyzed by the entire procedure.

6. Interferences

6.1 The following minerals have properties (that is, chemical or crystalline structure) which are very similar to asbestos minerals and may interfere with the analysis by causing false positives to be recorded during the test. Therefore, literature references for these materials must be maintained in the laboratory for comparison to asbestos minerals so that they are not misidentified as asbestos minerals.

6.1.1 *Antigorite.*
6.1.2 *Palygorskite (Attapulgite).*
6.1.3 *Halloysite.*
6.1.4 *Pyroxenes.*
6.1.5 *Sepiolite.*
6.1.6 *Vermiculite scrolls.*
6.1.7 *Fibrous talc.*
6.1.8 *Hornblende* and other amphiboles not listed in 5.1.3.

6.2 Collection of any dust particles greater than 1 mm in size in this test method may cause an interference and, therefore, should be avoided.

7. Apparatus

7.1 *Transmission Electron Microscope (TEM)*, an 80 to 120 kV TEM, capable of performing electron diffraction, with a fluorescent screen inscribed with calibrated gradations, is required. The TEM must be equipped with energy dispersive X-ray spectroscopy (EDXA) and it must have a scanning transmission electron microscopy (STEM) attachment or be capable of producing a spot size of less than 250 nm in diameter at crossover.

7.2 *Energy Dispersive X-ray System (EDXA).*

7.3 *High Vacuum Carbon Evaporator*, with rotating stage.

7.4 *High Efficiency Particulate Air (HEPA)*, filtered negative flow hood.

7.5 *Exhaust or Fume Hood.*

7.6 *Particle-Free Water* (ASTM Type II, see Specification D 1193).

7.7 *Glass Beakers*, 50 mL.

7.8 *Glass Sample Containers*, with wide mouth screw cap (200 mL), or equivalent sealable container (height of the glass sample container should be approximately 13 cm high by 6 cm wide).

7.9 *Waterproof Markers.*

7.10 *Forceps (tweezers).*

7.11 *Ultrasonic Bath*, table top model (100 W, approximate, see 22.5).

7.12 *Graduated Pipettes*, 1, 5, and 10 mL sizes, glass or plastic.

7.13 *Filter Funnel*, 25 mm or 47 mm (either glass or disposable). Filter funnel assemblies, either glass or disposable plastic, and using either a 25 mm or 47 mm diameter filter.

7.14 *Side Arm Filter Flask*, 1000 mL.

7.15 *Mixed Cellulose Ester (MCE) Membrane Filters*, 25 or 47 mm diameter, ≤0.22 µm and 5 µm pore size.

7.16 *Polycarbonate (PC) Filters*, 25 or 47 mm diameter, ≤ 0.2 µm pore size.

7.17 *Storage Containers*, for the 25 or 47 mm filters (for archiving).

7.18 *Glass Slides.*

7.19 *Scalpel Blades.*

7.20 *Cabinet-type Desiccator*, or low temperature drying oven.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[8] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Acetone.*
8.3 *Dimethylformamide (DMF).*
8.4 *Chloroform.*
8.5 *1-methyl-2-pyrrolidone.*
8.6 *Glacial Acetic Acid.*
8.7 *Low Temperature Plasma Asher.*
8.8 *pH Paper.*
8.9 *Air Sampling Pump* (low volume personal-type pump).
8.10 *Rotameter.*
8.11 *Air Sampling Cassettes* (25 mm or 37 mm), containing 0.8 µm or smaller pore size MCE or PC filters.
8.12 *Cork Borer*, 7 mm.
8.13 *Non-Asbestos Mineral References*, as outlined in 6.1.
8.14 *Asbestos Standards*, as outlined in 3.1.2.
8.15 *Tygon*[9] *Tubing*, or equivalent.
8.16 *Vacuum Pump* (small), that can maintain a vacuum of approximately 92 kPa.
8.17 *Petri Dishes*, large, glass (approximately 90 mm in diameter).

[8] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[9] Tygon is a registered trademark of the DuPont Co.

8.18 *Stainless Steel or Aluminum Mesh Screen*, 30 to 40 mesh for Jaffe washer.

8.19 *Copper TEM Finder Grids*, 200 mesh.

8.20 *Carbon Evaporator Rods*.

8.21 *Lens Tissue*.

8.22 *Ashless Filter Paper*, 90 mm diameter, ASTM Class G (see Specification E 832).

8.23 *Reinforcement Rings*, gummed paper.

8.24 *Wash Bottles*, plastic.

8.25 *Reagent Alcohol*, HPLC grade (Fisher A995 or equivalent).

8.26 *Opening Mesh Screen*, plastic, 1.0 by 1.0 mm (Spectra-Mesh No. 146410 or equivalent).

8.27 *Static Neutralizer*.

8.28 *Muffle Furnace*, 480°C.

8.29 *Glazed Crucibles*.

8.30 *Jaffe Washer* **(4, 5)**.

8.31 *Diffraction Grating Replica*, with approximately 2160 lines/mm.

8.32 The following items are necessary if performing weight % calculations.

8.32.1 *Analytical Balance*, with readability of 0.01 mg or less. Exercise special care for the proper zeroing of the balance.

8.32.2 *Weighing Room*, with temperature and humidity control to allow weighing with an analytical balance to ±0.01 mg.

8.32.3 *Class M Weights*, for calibration of the analytical balance.

9. Sampling Procedure For Microvacuum Technique

9.1 For sampling asbestos-containing dust in either indoor or outdoor environments, commercially available cassettes must be used. Cassettes and sampling nozzles must be new and not previously used. Air monitoring cassettes containing 25 mm or 37 mm diameter mixed cellulose ester (MCE) or polycarbonate (PC) filter membranes with a pore size less than or equal to 0.8 µm are required. The number of samples collected depends upon the specific circumstances of the study.

9.2 Maintain a log of all pertinent sampling information and sampling locations.

9.3 Sampling pumps and flow indicators shall be calibrated using a certified standard apparatus or assembly (see Practice D 3195).

9.4 Record all calibration information **(6)**.

9.5 Perform a leak check of the sampling system at each sampling site by activating the pump with the closed sampling cassette in line. Any air flow shows that a leak is present that must be eliminated before initiating the sampling operation.

9.6 Attach the sampling cassette to the sampling pump at the outlet side of the cassette with plastic tubing (see 8.15). The plastic tubing shall be long enough that sample areas can be reached without interference from the sampling pump. Attach a clean, approximately 25.4 mm long, piece of plastic tubing (6.35 mm internal diameter) directly to the inlet orifice. Use this piece of tubing as the sampling nozzle. Cut the sampling end of the tubing at a 45° angle as illustrated in Fig. 1. The exact design of the nozzle is not critical as long as some vacuum break is provided to avoid simply pushing the dust around on the surface with the nozzle rather than vacuuming it into the cassette. The internal diameter of the nozzle and flow rate of the pump may vary as long as the air velocity within the body of the nozzle is 100 (±10) cm/s. This air velocity can be achieved with an internal sampling tube diameter of 6.35 mm (¼ in.) and a flow rate of 2 L/min.

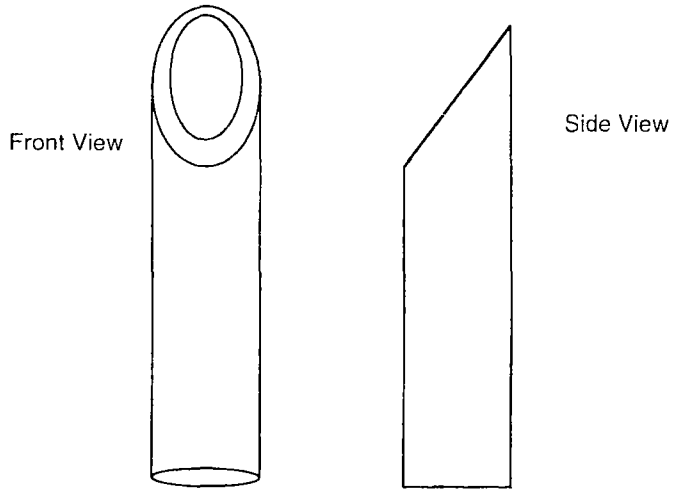

FIG. 1 Sampling End of Tubing

9.7 Delineate and measure the surface area of interest. A sample area of 100 cm^2 is vacuumed until there is no visible dust or particulate remaining. Perform a minimum of two orthogonal passes on the surface with a minimum of 2 min of sampling time. Avoid scraping or abrading the surface being sampled. (Do not sample any debris or dust particles greater than 1 mm in diameter (see 5.2)). Smaller or larger areas can be sampled, if needed. For example, some surfaces of interest may have a smaller area than 100 cm^2. Less dusty surfaces may require vacuuming of larger areas. Unlike air samples, the overloading of the cassettes with dust will not be a problem. As defined in 3.2.5, only dust shall be collected for this analysis.

9.8 At the end of the sample collection, invert the cassette so that the nozzle inlet faces up before shutting off the power to the pump. The nozzle is then sealed with a cassette end plug and the cassette/nozzle taped or appropriately packaged to prevent separation of the nozzle and cassette assembly. A second option is the removal of the nozzle from the cassette, then plugging of the cassette and shipment of the nozzle (also plugged at both ends) sealed in a separate closeable plastic bag. A third option is placing the nozzle inside the cassette for shipment. The nozzle is always saved to be rinsed because a significant percentage of the dust drawn from a lightly loaded surface may adhere to the inside walls of the tubing.

9.9 Check that all samples are clearly labeled, that all dust sampling information sheets are completed, and that all pertinent information has been enclosed, in accordance with laboratory quality control practices, before transfer of the samples to the laboratory. Include an unused cassette and nozzle as a field blank.

9.10 Wipe off the exterior surface of the cassettes with disposable wet towels or "baby wipes" prior to packaging for shipment.

10. Sample Shipment

10.1 Ship dust samples to an analytical laboratory in a

sealed container, but separate from any bulk or air samples. The cassettes must be tightly sealed and packed in a material free of fibers or dust to minimize the potential for contamination. Plastic bubble pack is probably the most appropriate material for this purpose.

11. Sample Preparation

11.1 Under a negative flow HEPA hood, carefully wet-wipe the exterior of the cassettes to remove any possible contamination before taking cassettes into a clean preparation area.

11.2 Perform sample preparation in a clean facility that has a separate work area from both the bulk and air sample preparation areas.

11.3 Initial specimen preparation shall take place in a clean HEPA filtered negative pressure hood to avoid any possible contamination of the laboratory or personnel, or both, by the potentially large number of asbestos structures in an asbestos-containing dust sample. Cleanliness of the preparation area hoods is measured by the cumulative process blank concentrations (see Section 12).

11.4 If a weight % determination is reported, pre-weigh the laboratory filter in accordance with this section, otherwise, proceed with 11.5.

11.4.1 Dry the laboratory filter under vacuum in a vacuum desiccator for at least 15 min.

11.4.2 Release the vacuum, remove the desiccator cover, and equilibrate the laboratory filters in the weighing room for at least 1 h.

11.4.3 Weigh the laboratory filter and record the filter tare weight, W1 (mg).

11.4.3.1 Zero the balance before each weighing.

11.4.3.2 Handle the filter with forceps.

11.4.3.3 Pass the filter over an antistatic radiation source. Repeat this step if the filter does not release easily from the forceps or if the filter attracts the balance pan.

11.4.4 Allow the laboratory filter to stand in the weighing room for an additional 8 to 16 h.

11.4.5 Reweigh the laboratory filter. If the second weight differs by more than 0.01 mg from the first tare weight (W1), discard the filter.

11.5 All sample preparation steps 11.5.1-11.5.6 shall take place in the dust preparation area inside a HEPA hood.

11.5.1 Remove the upper plug from the sample cassette and container and carefully introduce approximately 10 mL of a 50/50 mixture of particle-free water and reagent alcohol into the cassette using a plastic wash bottle. If the plugged nozzle was left attached to the cassette, then remove the plug and introduce the water/alcohol into the cassette through the tubing, and then remove the tubing if it is visibly clean.

11.5.2 Replace the upper plug or the sample cap, and lightly shake the dust suspension by hand for 3 s.

11.5.3 Remove the entire cap of the cassette and pour suspension through a 1.0 by 1.0 mm opening screen into a pre-cleaned 200 mL glass specimen bottle. All visible traces of the sample contained in the cassette shall be rinsed onto the screen with a plastic wash bottle containing the 50/50 mixture of water and alcohol. Repeat this procedure two additional times for a total of three washings. Next, rinse the nozzle two or three times through the screen into the specimen bottle with the 50/50 mixture of water and alcohol. Typically, the total amount of the 50/50 mixture used in the rinse is 50 to 75 mL. Remove the 1.0 by 1.0 mm screen and discard.

11.5.4 Use either a disposable plastic filtration unit or a glass filtering unit for filtration of the suspension. If a weight % determination is to be reported, use pre-weighed laboratory filters; see 11.4.

11.5.4.1 If a disposable plastic filtration unit is used, unwrap a new disposable plastic filter funnel unit (either 25 or 47 mm diameter) and remove the tape around the base of the funnel. Remove the funnel and discard the top filter supplied with the apparatus, while retaining the coarse polypropylene support pad in place. Assemble the unit with the adapter and a properly sized neoprene stopper, and attach the funnel to the 1000-mL side-arm vacuum flask. Place a 5.0 μm pore size MCE (backing filter) on the support pad. Wet it with a few mL of particle-free water and place an MCE or PC filter (≤0.2 μm pore size) on top of the backing filter. Apply a vacuum, ensuring that the filters are centered and pulled flat without air bubbles. Any irregularities on the filter surface requires discard of that filter. After the filter has been seated properly, replace the funnel, and reseal it with the tape. Return the flask to atmospheric pressure.

11.5.4.2 If a glass filtration unit is used, place a 5 μm pore size MCE (backing filter) on the glass frit surface. Wet the filter with particle-free water, and place an MCE or PC filter (≤0.2 μm pore size) on top of the backing filter. Apply a vacuum, ensuring that the filters are centered and pulled flat without air bubbles. Replace the filters if any irregularities are seen on the filter surface. Before filtration of the sample suspension, prepare a blank filter by filtration of 50 mL of particle-free water. After completion of the filtration, do not allow the filtration funnel assembly to dry because contamination is then more difficult to remove. Wash any residual suspension from the filtration assembly by holding it under a flow of water, then rub the surface with a clean paper towel soaked in a detergent solution. Repeat the cleaning operation, and then rinse several times in particle-free water.

11.5.5 Pour the suspension into the filter funnel. Rinse the beaker into the funnel at least three times, using distilled water.

11.5.6 Apply vacuum to the flask and draw the mixture through the filter.

11.5.7 Disassemble the filtering unit and carefully remove the sample filter with fine tweezers. Place the completed sample filter particle side up, into a precleaned, labeled disposable plastic petri dish or other similar container.

11.6 If a weight % determination is reported, weigh the filter and solids using the method described in this section.

11.6.1 Place the filter into a low temperature drying oven (60°C) for 1 h.

11.6.2 Remove the filter and allow the filter to equilibrate in the weighing room for at least 1 h.

11.6.3 Weigh the filter and record the filter weight, W2 (mg).

11.6.3.1 Zero the balance before each weighing.

11.6.3.2 Handle the filter with forceps.

11.6.3.3 Pass the filter over an antistatic radiation source. Repeat this step if the filter does not release easily from the forceps or if the filter attracts the balance pan.

11.6.3.4 Use the same balance for weighing the filters before and after filtration of the suspension.

11.7 Place the filter or a measured portion of the filter into a glazed crucible and cover with an appropriate lid.

11.7.1 Pre-heat the muffle furnace to 480°C. Regulate this temperature at ±5°C throughout the ashing of the sample.

11.7.2 Place the crucible in the muffle furnace. Ash the sample for 12 h, or until the weight has stabilized.

11.7.3 Remove the crucible from the muffle furnace and allow it to cool.

11.8 Rinse the ash from the crucible into a 200 mL glass specimen bottle. Rinse all visible traces of the sample contained in the crucible into the specimen bottle using a plastic wash bottle containing particle-free water. This procedure is repeated two additional times for a total of three washings.

11.8.1 Bring the volume of the suspension up to 100 mL using distilled water.

11.8.2 Adjust the pH of the suspension to 3 to 4 by using a 10.0 % solution of acetic acid. Use pH paper for testing. Filter the suspension within 24 h to avoid problems associated with bacterial and fungal growth.

11.9 Use either a disposable plastic filtration unit or a glass filtering unit for filtration of portions of the suspension. The ability of an individual filtration unit to produce a uniformly loaded filter may be tested by filtration of a colored particulate suspension such as carbon black.

11.9.1 If a disposable plastic filtration unit is used then unwrap a new disposable plastic filter funnel unit (either 25 or 47 mm diameter) and remove the tape around the base of the funnel. Remove the funnel and discard the top filter supplied with the apparatus, while retaining the coarse polypropylene support pad in place. Assemble the unit with the adapter and a properly sized neoprene stopper, and attach the funnel to the 1000 mL side arm vacuum flask. Place a 5.0 µm pore size MCE (backing filter) on the support pad. Wet it with a few mL of particle-free water and place an MCE or PC filter (≤0.2 µm pore size) on top of the backing filter. Apply a vacuum, ensuring that the filters are centered and pulled flat without air bubbles. Any irregularities on the filter surface dictates discarding of that filter. After the filter has been seated properly, replace the funnel and reseal it with the tape. Return the flask to atmospheric pressure.

11.9.2 If a glass filtration unit is used, place a 5 µm pore size MCE (backing filter) on the glass frit surface. Wet the filter with particle-free water, and place an MCE or PC filter (≤0.2 µm pore size) on top of the backing filter. Apply a vacuum, ensuring that the filters are centered and pulled flat without air bubbles. Replace the filters if any irregularities are seen on the filter surface. Before filtration of each set of sample portions, prepare a blank filter by filtration of 50 mL of particle-free water. If portions of the same sample are filtered in order of increasing concentration, the glass filtration unit need not be washed between filtrations. After completion of the filtration, do not allow the filtration funnel assembly to dry because contamination is then more difficult to remove. Wash any residual suspension from the filtration assembly by holding it under a flow of water, then rub the surface with a clean paper towel soaked in a detergent solution. Repeat the cleaning operation, and then rinse several times in particle-free water.

11.9.3 With the flask at atmospheric pressure, add 20 mL of particle-free water into the funnel. Cover the filter funnel with its plastic cover if the disposable filtering unit is used.

11.10 Briefly hand shake (3 s) the capped bottle with the sample suspension, then place it in a tabletop ultrasonic bath and sonicate for 15 min. Maintain the water level in the sonicator at the same height as the solution in the sample bottle. Carry out preparation steps 11.10-11.11.5 in a HEPA hood.

11.10.1 Calibrate the ultrasonic bath in accordance with 22.5. The ultrasonic bath must be operated at equilibrium temperature.

11.11 Estimate the amount of liquid to be withdrawn to produce an adequate filter preparation. Experience has shown that a light staining of the filter surface will yield a suitable preparation for analysis. Filter at least 1.0 mL, but no more than half the total volume. Draw this portion from the suspension while it is in the sonicator. If after examination in the TEM, the smallest volume filtered (1.0 mL) yields an overloaded sample, perform additional serial dilutions of the suspension.

11.11.1 If serial dilutions are required, repeat step 11.10 before the serial dilution portion is taken. The recommended procedure for a serial dilution is to mix 10 mL of the sample solution with 90 mL of particle-free water in a clean sample bottle to obtain a 1:10 serial dilution. Follow good laboratory practices when performing serial dilutions.

11.11.2 Insert a new disposable pipette halfway into the sample suspension and withdraw a portion. Avoid pipetting any of the large floating or settled particles. Uncover the filter funnel and dispense the mixture from the pipette into the water in the funnel.

11.11.3 Apply vacuum to the flask and draw the mixture through the filter.

11.11.4 Discard the pipette.

11.11.5 Disassemble the filtering unit and carefully remove the sample filter with fine tweezers. Place the completed sample filter, particle side up, into a precleaned, labeled, disposable, plastic petri dish or other similar container.

11.11.6 In order to ensure that an optimally-loaded filter is obtained, it is recommended that filters be prepared from several different portions of the dust suspension. For this series of filters, it is recommended that the volume of each portion of the original suspension be a factor of five higher than the previous one. If the filters are prepared in order of increasing portion volume, all of the filters for one sample can be prepared using one plastic disposable filtration unit, or without cleaning of glass filtration equipment between individual filtrations.

11.11.7 There are many practical methods for drying MCE filters; the following are two examples that can be used: (*1*) dry MCE filters for at least 12 h (over desiccant) in an airtight cabinet-type desiccator; (*2*) to shorten the drying time (if desired), remove a plug of the damp filter and attach to a glass slide in accordance with 13.1.2 and 13.1.3. Place the slide with a filter plug or filter plugs (up to eight plugs can be attached to one slide) on a bed of desiccant, in the desiccator for 1 h.

11.11.8 PC filters do not require lengthy drying before

preparation, but may be placed in a desiccator for at least 30 min before preparation.

11.12 Prepare TEM specimens from small sections of each dried filter using the appropriate direct transfer preparation method.

12. Blanks

12.1 Prepare sample blanks that include both a process blank (50 mL of ultrapure water) for each set of samples analyzed and one unused filter from each new box of sample filters (MCE or PC). If glass filtering units are used, prepare and analyze a process blank each time the filtering unit is cleaned. For example, if one glass filtering unit is used for ten different individual samples, then ten process blanks are prepared and analyzed. Blanks will be considered contaminated if, after analysis, they are shown to contain more than 53 asbestos structures per square millimetre. This generally corresponds to three or four asbestos structures found in ten grid openings. The source of the contamination must be found before any further analysis can be performed. Reject samples that were processed along with the contaminated blanks and prepare new samples after the source of the contamination is found.

12.2 Prepare field blanks which are included with sample sets in the same manner as the samples, to test for contamination during the sampling, shipping, handling, and preparation steps of the method.

13. TEM Specimen Preparation of Mixed Cellulose Ester (MCE) Filters

NOTE 1—Use of either the acetone or the DMF-acetic acid method is acceptable.

13.1 *Acetone Fusing Method*:

13.1.1 Remove a section (plug) from any quadrant of the sample and blank filters. Sections can be removed from the filters using a 7 mm cork borer. The cork borer must be wet wiped after each time a section is removed.

13.1.2 Place the filter section (particle side up) on a clean microscope slide. Affix the filter section to the slide with a gummed page reinforcement or other suitable means. Label the slide with a glass scribing tool or permanent marker.

13.1.3 Prepare a fusing dish from a glass petri dish and a metal screen bridge. To prepare the fusing dish, make a pad from five to six ashless paper filters and place in the bottom of the petri dish. Place the screen bridge on top of the pad and saturate the filter pads with acetone. Place the slide on top of the bridge in the petri dish and cover the dish. Wait approximately 5 min for the sample filter to fuse and clear.

13.2 *DMF-Acetic Acid Method*:

13.2.1 Place a drop of clearing solution that consists of 35 % dimethylformamide (DMF), 15 % glacial acetic acid, and 50 % Type II water (v/v) on a clean microscope slide. Gauge the amount used so that the clearing solution just saturates the filter section.

13.2.2 Carefully lay the filter segment, sample surface upward, on top of the solution. Bring the filter and solution together at an angle of about 20° to help exclude air bubbles. Remove any excess clearing solution. Place the slide in an oven or on a hot plate, in a fume hood, at 65 to 70°C for 10 min.

13.3 Plasma etching of the collapsed filter is required.

13.3.1 The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher. Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the exact conditions that must be used. Insufficient etching will result in a failure to expose embedded fibers, and too much etching may result in the loss of particles from the filter surface. To determine the optimum time for ashing, place an unused 25 mm diameter MCE filter in the center of a glass microscope slide. Position the slide approximately in the center of the asher chamber. Close the chamber and evacuate to a pressure of approximately 4 Pa, while admitting oxygen to the chamber at a rate of 8 to 20 cm^3/min. Adjust the tuning of the system so that the intensity of the plasma is maximized. Determine the time required for complete oxidation of the filter. Adjust the system parameters to achieve complete oxidation of the filter in a period of approximately 15 min. For etching of collapsed filters, use these operating parameters for a period of 8 min. For additional information on calibration, see the AHERA **(4)**, NISTIR **(7)**, or ISO/10312 and ISO/CD 13794.

13.3.2 Place the glass slide containing the collapsed filters into a low-temperature plasma asher, and etch the filter.

13.4 Carbon coating of the collapsed and etched filters is required.

13.4.1 Carbon coating must be performed with a high-vacuum coating unit, capable of less than 10^{-4} torr (13 mPa) pressure. Units that are based on evaporation of carbon filaments in a vacuum generated only by an oil rotary pump have not been evaluated for this application and must not be used. Carbon rods used for evaporators shall be sharpened with a carbon rod sharpener to necks of about 4-mm length and 1-mm diameter. The rods are installed in the evaporator in such a manner that the points are approximately 100 to 120 mm from the surface of the microscope slide held in the rotating device.

13.4.2 Place the glass slide holding the filters on the rotation device, and evacuate the evaporator chamber to a pressure of at most 13 mPa. Perform the evaporation in very short bursts, separated by 3 to 4 s to allow the electrodes to cool. An alternate method of evaporation is accomplished by using a low continuous applied current. An experienced analyst can judge the thickness of the carbon film to be applied. Some tests should be made first on unused filters. If the carbon film is too thin, large particles will be lost from the TEM specimen, and there will be few complete and undamaged grid openings on the specimen. If the coating is too thick, it will lead to a TEM image that is lacking in contrast, and the ability to obtain electron diffraction patterns will be compromised. The carbon film should be as thin as possible and still remain intact on most of the grid openings of the TEM specimen.

13.5 *Preparation of the Jaffe Washer*:

13.5.1 The precise design of the Jaffe washer is not considered important, so any one of the published designs may be used **(5, 8)**. One such washer consists of a simple stainless steel

bridge contained in a glass petri dish, as illustrated in Fig. 2.

13.5.2 Place several pieces of lens tissue on the stainless steel bridge. The pieces of lens tissue shall be large enough to completely drape over the bridge and into the solvent. In a fume hood, fill the petri dish with acetone (or DMF) until the height of the solvent is brought up to contact the underside of the metal bridge.

13.6 *Placing the Specimens into the Jaffe Washer:*

13.6.1 Place the TEM grids shiny side up on a piece of lens tissue or filter paper so that individual grids can be easily picked up with tweezers.

13.6.2 Prepare three grids from each sample.

13.6.2.1 Using a curved scalpel blade, excise at least two square (3 mm by 3 mm) pieces of the carbon-coated MCE filter from the glass slide.

13.6.2.2 Place the square filter piece carbon-side up on top of a TEM specimen grid.

13.6.2.3 Place the whole assembly (filter/grid) on the saturated lens tissue in the Jaffe washer.

13.6.2.4 Place the three TEM grid sample filter preparations on the same piece of lens tissue in the Jaffe washer.

13.6.2.5 Place the lid on the Jaffe washer and allow the system to stand for several hours.

13.7 Alternately, place the grids on a low level (petri dish filled to the ⅛ mark) DMF Jaffe washer for 60 min. Add enough solution of equal parts DMF/acetone to fill the washer to the screen level. Remove the grids after 30 min if they have cleared, that is, all filter material has been removed from the carbon film, as determined by inspection in the TEM.

13.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean marked grid box.

14. TEM Specimen Preparation of Polycarbonate (PC) Filter

14.1 Cover the surface of a clean microscope slide with two strips of double-sided adhesive tape.

14.2 Cut a strip of filter paper slightly narrower than the width of the slide. Position the filter paper strip on the center of the length of the slide.

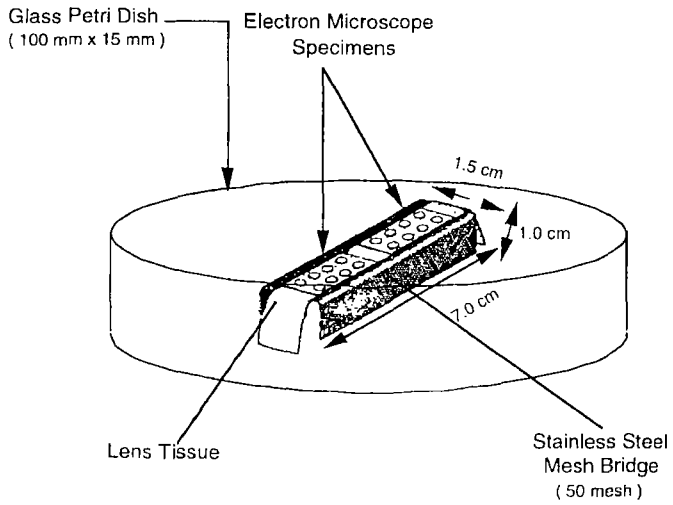

FIG. 2 Design of Solvent Washer (Jaffe Washer)

14.3 Using a clean, curved scalpel blade, cut a strip of the PC filter approximately 25 by 6 mm. Use a rocking motion of the scalpel blade to avoid tearing the filter. Place the PC strip particle side up on the slide perpendicular to the long axis of the slide. The ends of the PC strip must contact the double sided adhesive tape. Each slide can hold several PC strips. With a glass marker, label (next to each PC strip) with the individual sample number.

14.4 Carbon coat the PC filter strips as discussed in 13.4.2. PC filters do not require etching.

NOTE 2—**Caution:** Do not overheat the filter sections while carbon coating.

14.5 Prepare a Jaffe washer as described in 13.5, but fill the washer with chloroform or 1-methyl-2-pyrrolidone to the level of the screen.

14.6 Using a clean curved scalpel blade, excise three, 3 mm square filter pieces from each PC strip. Place the filter squares carbon side up on the shiny side of a TEM grid. Pick up the grid and filter section together and place them on the lens tissue in the Jaffe washer.

14.7 Place the lid on the Jaffe washer and leave the grids for at least 4 h. Best results are obtained with longer wicking times, up to 12 h.

14.8 Carefully remove the grids from the Jaffe washer. Allow the grids to dry before placing them in a clean, marked grid box.

15. Grid Opening Measurements

15.1 TEM grids must have a known grid opening area. Determine this area as follows:

15.1.1 Measure at least 20 grid openings in each of 20 random 75 μm (200-mesh) copper grids for a total of 400 grid openings for every 1000 grids used, by placing the 20 grids on a glass slide and examining them under the optical microscope. Use a calibrated graticule to measure the average length and width of the 20 openings from each of the individual grids. From the accumulated data, calculate the average grid opening area of the 400 openings.

15.2 Grid area measurements can also be made at the TEM at a calibrated screen magnification. Typically measure one grid opening for each grid examined. Measure grid openings in both the x and y directions and calculate the area.

15.3 Pre-calibrated TEM grids are also acceptable for this test method.

16. TEM Method

16.1 Calibrate the microscope routinely for magnification, camera constant, and EDXA settings (see Section 22 for more detailed discussion).

16.2 Microscope settings: 80 to 120 kV, 15 000 to 20 000× screen magnification for analysis as determined in 16.7.

16.3 Analyze two grids for each sample. Analyze approximately one-half of the required grid openings on one sample grid preparation and the remaining half on a second grid preparation. See 16.7 and 17.10 for the number of grid openings which must be analyzed.

16.4 *Determination of Specimen Suitability:*

16.4.1 Carefully load the TEM grid, carbon side facing up (in the TEM column) with the grid bars oriented parallel/

perpendicular to the length of the specimen holder. Use a hand lens or loupe, if necessary. This procedure will line up the grid with the *x* and *y* translation directions of the microscope. Insert the specimen holder into the microscope.

16.4.2 Scan the entire grid at low magnification (250× to 1000×) to determine its suitability for high magnification analysis as specified in 16.4.3.

16.4.3 Grids are acceptable for analysis if the following conditions are met:

16.4.3.1 The fraction of grid openings covered by the replica section is at least 50 %.

16.4.3.2 Relative to that section of the grid covered by the carbon replica, the fraction of intact grid openings is greater than 50 %.

16.4.3.3 The fractional area of undissolved filter is less than 10 %.

16.4.3.4 The fraction of grid openings with overlapping or folded replica film is less than 50 %.

16.4.3.5 At least 20 grid openings that have no overlapping or folded replica, are less than 5 % covered with holes and have less than 5 % opaque area due to incomplete filter dissolution.

16.5 *Determination of Grid Opening Suitability*:

16.5.1 If the grid meets acceptance criteria, choose a grid opening for analysis from various areas of the grid so that the entire grid is represented. Determine the suitability of each individual grid opening to be analyzed prior to the analysis.

16.5.2 The individual grid opening must have less than 5 % holes over its area.

16.5.3 Grid openings must be less than 25 % covered with particulate matter.

16.5.4 Grid openings must be uniformly loaded.

16.6 Observe and record the orientation of the grid, at 80 to 150×, on a grid map record sheet along with the location of the grid openings that are examined for the analysis. If indexed grids are used, a grid map is not required, but the identifying coordinates of the grid square must be recorded.

16.7 At low magnification (1000×), scan approximately 50 grid openings to find the largest width of an asbestos fiber or bundle. If an asbestos fiber or bundle is observed, adjust the microscope magnification so that 1 mm on the fluorescent screen corresponds to approximately 10 % of the width of the previously selected large structure. Examine the sample at the lower of this magnification or 20 000×. If no fibers or bundles are observed during the low magnification scan, arbitrarily choose one grid opening. Begin high magnification (15 000 to 20 000×) scans with this grid opening.

16.7.1 Record the dimensions of the structure with the largest width. Calculate the mass of this structure (see Section 17).

16.7.2 Continue scanning at the appropriate magnification until either the mass of the large structure selected in 16.7.1 is no greater than 1/10 of the total asbestos mass observed or the stopping rules (17.10) apply.

17. Data Recording Rules

17.1 Record on the count sheet (Fig. 3) any continuous grouping of particles in which an asbestos fiber is detected. Classify asbestos structures as fibers, bundles, clusters, or matrices as defined in 3.2.

17.2 Use the AHERA criteria for fiber, bundle, cluster, and matrix identification **(4)**. Record length and width measurements.

17.2.1 For bundles and fibers, record the length and width of the structure.

17.2.2 For disperse clusters, record the overall length and width of the structure. Record, in order of decreasing length, up to five component fibers or bundles in accordance with 17.2.1. These component fibers or bundles are not counted in determining overall structure count or stopping rules.

17.2.3 For compact clusters, record the overall length and width of the structure.

17.2.4 For disperse matrices, record the overall length and width of the structure. Record, in order of decreasing length, up to five component fibers or bundles in accordance with 17.2.1. These component fibers or bundles are not counted in determining overall structure count or stopping rules.

17.2.5 For compact matrices, record the overall length and width of the structure.

17.2.6 Calculate the mass of an individual structure as shown in 20.1.

17.3 Record NSD (No Structures Detected) when no structures are detected in the grid opening.

17.4 Identify structures classified as chrysotile identified by either electron diffraction or X-ray analysis and record on a count sheet. Verify at least one out of every ten chrysotile structures by X-ray analysis.

17.5 Structures classified as amphiboles by X-ray analysis and electron diffraction are recorded on the count sheet. For more information on identification, see Yamate, et al **(5)** or Chatfield and Dillon **(8)**.

17.6 Record a typical electron diffraction pattern for each type of asbestos observed for each group of samples (or a minimum of every five samples) analyzed. Record the micrograph number on the count sheet. Record at least one X-ray spectrum for each type of asbestos observed per sample. Attach the print-outs to the back of the count sheet. If the X-ray spectrum is stored, record the file and disk number on the count sheet.

17.7 *Counting Rules*:

17.7.1 At a screen magnification of between 15 000 and 20 000× evaluate the grids for the most concentrated sample loading; reject the sample if it is estimated to contain more than 50 asbestos structures per grid opening. Proceed to the next lower concentrated sample until a set of grids are obtained that have less than 30 asbestos structures per grid opening.

17.8 *Analytical Sensitivity*—An absolute analytical sensitivity specified in terms of mass cannot be defined for this test method. A nominal analytical sensitivity for an analysis can be defined on the basis of the smallest asbestos fibril reportable by this test method, 0.5 μm by 0.025 μm. Assuming the following values for the parameters defined in Section 19: EFA = 1150 mm^2; GO = 10; GOA = 0.006 mm^2; SPL = 100 cm^2; V = 50 mL, this equates to an analytical sensitivity of 0.24 pg of asbestos per square centimetre. This sensitivity can be improved by increasing the amount of liquid filtered, increasing the number of grid openings analyzed, or decreasing the size of the final filter.

Page # ______ / ______

Client ______	Microscope ______	# Grid Openings ______
Sample ID ______	Magnification ______	# Asbestos ______
Job Number ______	Accelerating Voltage ______	# Nonasbestos ______
Average Grid Opening, mm ______	Operator ______	# Asbestos ≥ 5 μm ______
Date Analyzed ______	EDS Disk # ______	# Nonasbestos ≥ 5 μm ______
	Position ______	

Field	Fiber	Length μm	Width, μm	Fiber Type			Morph	Mass	EDS	Photo	SAED	Amp ID	√ by
				Amp	Chr	Nas							
				Amp	Chr	Nas							
				Amp	Chr	Nas							
				Amp	Chr	Nas							
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				Amp	Chr	Nas							
				Amp	Chr	Nas							

Amphibole Abbreviations: Act-Actinolite, Ant-Anthophyllite, Cro-Crocidolite, Gru-Grunerite (Amosite), Tre-Tremolite.

FIG. 3 Count Sheet

17.9 *Limit of Detection*—Because the Limit of Detection is dependent on the counting of structures, the Limit of Detection for this test method is defined as less than four structures measured during the analysis. If no asbestos structures are observed, the Limit of Detection is four times the analytical sensitivity calculated assuming fiber dimensions of 0.5 μm by 0.025 μm (see 17.8). If more than four structures are detected, the Limit of Detection equals the mass calculated based on the measurements of the four smallest structures reported. If less than four structures are observed, the Limit of Detection is the calculated value of concentration based on four times the average mass of the structures observed.

17.10 *Stopping Rules*:

17.10.1 Continue analyzing the sample until the mass of the largest asbestos structure (16.7) accounts for 10 % of the total asbestos mass counted or until the total number of grid squares analyzed is either 100 or 10 × 20 000/selected magnification, whichever is smaller.

17.10.1.1 Complete the analysis of the grid square in which the stopping rules are satisfied.

17.10.2 If more than 10 % of the counted structures are either compact clusters or compact matrices, then the sample must be reprepared starting with 11.10, using either longer ultrasonic treatment or an ultrasonic probe to disperse the complex structures.

17.11 After analysis, remove the grids from the TEM, and replace them in the appropriate grid storage holder.

18. Sample Storage

18.1 The washed-out sample cassettes can be discarded after use.

18.2 Sample grids and unused filter sections must be stored for a minimum of one year.

19. Calculations

19.1 *Calculation of Results*—Use the following equation to determine the amount of asbestos in the sample:

$$\text{asbestos, g/cm}^2 = \frac{EFA \times 100\ \text{mL} \times M}{GO \times GOA \times V \times SPL \times FF} \quad (1)$$

where:

M = total asbestos mass counted, g,
EFA = effective filter area of the final sampling filter, mm^2,
GO = number of grid openings counted,
GOA = average grid opening area, mm^2,
SPL = surface area sampled, cm^2,
V = volume of sample, mL, filtered in step 11.5.9, representing the actual volume taken from the original 100 mL suspension, and
FF = fraction of filter ashed in 11.7.

19.1.1 If the mass of asbestos per mass of analyzed dust is to be reported, calculate the weight % as follows: Sum the mass of asbestos from 20.1 to obtain a total mass of asbestos. Estimate the weight % using the following equation:

$$\text{asbestos, weight percent} = \frac{EFA \times 100\ \text{mL} \times M \times 100\ \%}{GO \times GOA \times V \times FF \times (W_2 - W_1)} \quad (2)$$

where:

W_1 = pre-weight of the filter, 11.4.1, mg, and
W_2 = weight of redeposit filter after the suspension has been filtered, as in 11.6.1, mg.

20. Reporting

20.1 Mass for each asbestos structure is calculated using the methods described in ISO/CD 13794.

20.1.1 The mass of a fiber or bundle is calculated as:

$$\text{Mass}_{\text{fiber}}(\text{g}) = \frac{\pi}{4} \times L \times W^2 \times D \times 10^{-12} \quad (3)$$

where:

L = length of the fiber, μm
W = width of the fiber, μm, and
D = density of fiber, Mg/m^3 **(1)**, **(2)**, or **(3)**.

20.1.1.1 The following densities are used:

Mineral	Density, Mg/m^3
Chrysotile	2.55
Crocidolite	3.37
Amosite	3.43
Anthophyllite	3.00
Tremolite	3.00
Actinolite	3.10

20.1.2 The mass of a disperse matrix or a disperse cluster is estimated by summing the mass of the component fibers or bundles. The mass of a compact cluster or compact matrix is not determined.

20.2 Report the following information for each dust sample analyzed:

20.2.1 Types of asbestos present,
20.2.2 Number of asbestos structures present,
20.2.3 Concentration in mass per cm^2 for each asbestos type present,
20.2.4 The analytical sensitivity,
20.2.5 Effective filtration area,
20.2.6 Average size of the TEM grid openings that were counted,
20.2.7 Number of grid openings examined,
20.2.8 Sample dilution used,
20.2.9 Area of the surface sampled,
20.2.10 Listing of size data for each structure counted, and
20.2.11 A copy of the TEM count sheet for each sample or a complete listing of the raw data.

21. Quality Control/Quality Assurance

21.1 In general, the laboratory's quality control checks are used to verify that a system is performing according to specifications regarding accuracy and consistency. In an analytical laboratory, spiked or known quantitative samples are normally used. However, due to the difficulties in preparing known quantitative asbestos samples, routine quality control testing focuses on re-analysis of samples (duplicate recounts).

21.1.1 Re-analyze samples at a rate of 1/10 of the sample sets (1 out of every 10 samples analyzed not including laboratory blanks). The re-analysis shall consist of a second sample preparation obtained from the final filter.

21.1.2 In addition, quality assurance programs must follow the criteria shown in the Federal Register for AHERA samples **(4)** and in the NISTIR document **(7)**. These documents describe sample custody, sample preparation, blank checks for contamination, calibration, sample analysis, analyst qualifications, and technical facilities.

22. Calibrations

22.1 Perform calibrations of the instrumentation on a regular basis, and retain a calibration record for each TEM in the laboratory, in accordance with the laboratory's quality assurance program.

22.2 Record calibrations in a log book along with dates of calibration and the attached backup documentation.

22.3 A calibration list for the instrument is as follows:

22.3.1 *TEM*:

22.3.1.1 Check the alignment and the systems operation. Refer to the TEM manufacturer's operational manual for detailed instructions.

22.3.1.2 Calibrate the camera length of the TEM in electron

diffraction (ED) operating mode before ED patterns of unknown samples are observed. Camera length can be measured by using a carbon coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thicker gold films will tend to mask weaker diffraction spots from the fibrous particles. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings from thicker films are unnecessary. Alternatively, a gold standard specimen can be used to obtain an average camera constant calculated for that particular instrument that can then be used for ED patterns of unknowns taken during the corresponding period.

22.3.1.3 Perform magnification calibration at the fluorescent screen. This calibration must be performed at the magnification used for structure counting. Calibration is performed with a grating replica (for example, one containing at least 2160 lines/mm).

(a) (*a*) Define a field of view on the fluorescent screen. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).

(b) (*b*) Frequency of calibration will depend on the service history of the particular microscope.

(c) (*c*) Check the calibration after any maintenance of the microscope that involves adjustment of the power supply to the lens or the high-voltage system or the mechanical disassembly of the electron optical column (apart from filament exchange).

(d) (*d*) The analyst must ensure that the grating replica is placed at the same distance from the objective lens as the specimen.

(e) (*e*) For instruments that incorporate a eucentric tilting specimen stage, all specimens and the grating replica must be placed at the eucentric position.

22.3.1.4 The smallest spot size of the TEM must be checked.

(a) (*a*) At the crossover point, photograph the spot size at a screen magnification of 15 000× to 20 000×. An exposure time of 1 s is usually adequate.

(b) (*b*) The measured spot size must be less than or equal to 250 nm.

22.4 *EDXA*:

22.4.1 The resolution and calibration of the EDXA must be verified.

22.4.1.1 Collect a standard EDXA Cu peak from the Cu grid.

22.4.1.2 Compare the X-ray energy versus channel number for the Cu peak and be certain readings are within ±10 eV.

22.4.2 Collect a standard EDXA of crocidolite asbestos (NIST SRM 1866).

22.4.2.1 The elemental analysis of the crocidolite must resolve the Na peak.

22.4.3 Collect a standard EDXA of chrysotile asbestos.

22.4.3.1 The elemental analysis of chrysotile must resolve both Si and Mg on a single chrysotile fiber.

22.5 Perform ultrasonic bath calibration as follows:

22.5.1 Fill the bath with water to a level equal to the height of suspension in the glass sample container that is used for the dust. Operate the bath until the water reaches the equilibrium temperature.

22.5.2 Place 100-mL of water (at approximately 20°C) in the 200-mL glass sample container, and record its temperature.

22.5.3 Place the sample container into the water in the ultrasonic bath (with the power turned off). After 60 s, remove the glass container and record its temperature.

22.5.4 Place 100-mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

22.5.5 Place the second sample container in the water in the ultrasonic bath (with the power turned on). After 60 s, remove the glass container and record its temperature.

22.5.6 Calculate the rate of energy deposition into the sample container using the following formula:

$$R = 4.185 \times \sigma \times \rho \times \frac{\theta_2 - \theta_1}{t} \quad (4)$$

where:

- 4.185 = joules/cal,
- R = energy deposition, MW/m^3,
- θ_1 = temperature rise with the ultrasonic bath not operating, °C,
- θ_2 = temperature rise with the ultrasonic bath operating, °C,
- t = time, s (to 60 s (22.5.3 and 22.5.5)),
- σ = specific heat of the liquid in the glass sample container (1.0 cal/g), and
- ρ = density of the liquid in the glass sample container (1.0 g/cm^3).

22.5.7 Adjust the operating conditions of the bath so that the rate of energy deposition is in the range of 0.08 to 0.12 MW/m^3, as defined by this procedure.

23. Precision and Bias

23.1 *Precision*—The precision of the procedure in this test method has not been determined. The failure to adequately disaggregate large structures, however, will decrease the precision of the results.

23.2 *Bias*—Bias of this test method has not been determined. The failure to adequately disaggregate large structures, however, will induce a negative bias to the results.

24. Keywords

24.1 asbestos; indirect; mass; microvacuuming; settled dust; TEM

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